OPTICAL FIBRE SENSOR FOR MEASURING THE DYNAMIC MECHANICAL PROPERTIES OF VISCOELASTIC SOLIDS

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Every bursted bubble has a glory! Each abysmal failure makes a point!

Every glowing path that goes astray, shows you how to find a better way.

So every time you stumble never grumble. Next time you’ll bumble even less!

For up from the ashes, up from the ashes, grow the roses of success!

_Roses of Success, Chitty Chitty Bang Bang_
Changes in the dynamic mechanical properties of human tissue can often be related to the health of the tissue, and hence can potentially be used in disease diagnosis. Although this is well recognised, at present there is limited information on the dynamic mechanical properties of tissue. It is thought that this is in part due to the difficulty in measuring these properties. Human tissue is a viscoelastic material and the dynamic mechanical properties of viscoelastic materials are frequency dependent. As such to fully understand the mechanical properties of tissue one must measure the complex Young’s modulus over a frequency range relevant to tissue palpation. This thesis explores the possibility of using an optical fibre-Bragg grating (FBG) sensor to measure the dynamic mechanical properties of viscoelastic materials. The FBG sensor system has been manufactured, calibrated and mechanically tested. Mechanical testing was performed by looking at the buckling and deflection forces acting on different lengths of the FBG sensor. These tests were performed to understand the effect of transverse and longitudinal forces on the sensor. The results showed that as the unsupported length of the FBG sensor increased, the force required to buckle and bend the sensor was reduced. A benchtop FBG sensor has been used to perform dynamic mechanical tests on polydimethylsiloxane (PDMS) samples with different ratios of cross linker (to mimic human tissues with different stiffness) over the frequency range of 0 - 20 Hz. The results show that the FBG sensor can be used to differentiate between the different ratios of PDMS. Samples of PDMS with crosslinker ratios of 10:1, 20:1, 40:1 and 50:1 were tested; the average absolute value of the complex moduli were 6.5 MPa, 3.4 MPa, 1.1 MPA and 0.9 MPa, respectively. A sample with different layers of PDMS was also tested to provide a better indication of inhomogeneous tissues found in the body. This analysis suggests that the spatial resolution of the sensor is comparable to the diameter of the optical fibre at approximately 0.5 mm. Finally, the results of initial efforts to develop a portable, compact actuator to turn these results into a useable diagnostic device are reported.
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DECLARATION

I declare that this thesis contains no material which has been accepted for the award of any other degree or diploma and to the best of my knowledge contains no work previously published or written by another author except where due reference is made in the text of this thesis.

Australia, May 2017

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ACRONYMS

AFM atomic force microscope
DMA dynamic mechanical analyser
FBG fibre-Bragg grating
FWHM full width half maximum
MRE magnetic resonance elastography
MRI magnetic resonance imaging
OCE optical coherence elastography
OCT optical coherence tomography
PDMS polydimethylsiloxane
PM phase mask
UV ultraviolet
INTRODUCTION

Changes in the dynamic mechanical properties of tissue can be caused by many health conditions [1]. Characterisation of the dynamic mechanical properties of tissue could therefore provide important information about the health of the tissue, potentially providing early diagnosis, improved treatment or leading to a better understanding of tissue properties or health [2]. For example, many cancers such as scirrhous carcinoma of the breast are stiffer than the surrounding tissue [3]. Currently there is limited data on how tissue properties change as they become diseased; this is important information if changes in tissue stiffness properties are to be used as an early indicator of poor tissue health and disease. This project aimed to undertake the fundamental work required for the creation of a sensor that can provide a quantitative measure of dynamic tissue properties. This sensor should be sufficiently sensitive that changes in tissue properties can be identified early and characterised. It should be small and portable so that it could be used in a variety of places, including the doctor’s rooms, at the patient bedside, during endoscopic examinations or during surgery. Another important characteristic of this sensor is the scale at which the changes in tissue properties are measured. The optimal scale for measuring these stiffness properties is the mesoscale. For the purpose of this thesis the mesoscale is defined as the range between the microscale (less than a few micrometres) and the macroscale (greater than a few millimetres). The microscale is better suited for fundamental studies of cell behaviour and is hard to apply in vivo. On the other hand, the macroscale may pick up changes later than desired. The macroscale is often related to changes in tissue properties in the later stages of disease onset, leading to fewer treatment options and poorer outcomes [4]. The mesoscale should allow potentially detrimental changes in tissue properties to be picked up early, aiding the chance of improved outcomes.

The sensor explored in this thesis is based on optical fibre. The most well-known use of optical fibres is in telecommunications applications; however they are also widely
utilised in sensing devices[5]. An optical fibre is as small as a single human hair. It is cheap, flexible and reliable, making it perfectly suited for a small and portable device that is endoscope compatible. As part of a dynamic tissue property characterisation sensor, it is envisaged that the optical fibre would be used as a sensitive force sensor, with the optical fibre pressed against the tissue being tested. Tissues with different stiffness properties should present different resistance forces in response to a given displacement of the optical fibre probe. Cancers of the bowel, stomach, nose, mouth and throat are commonly diagnosed using endoscopy. These types of cancers, that have restricted access, are perfectly suited for this type of optical fibre sensor.

Human tissue is a viscoelastic material and as such the tissue’s stiffness properties are frequency dependent, hence the need to characterise the dynamic properties. Water has similar frequency dependence. At low frequencies/speeds water is very soft; however if contact with water is made at higher speeds, for example falling off water skis, it can feel very hard, like concrete. As a result, in order to fully understand the stiffness properties of tissue and use them as a diagnostic, the optical fibre sensor must be applied to the tissue at a variety of frequencies thus creating a frequency response or stiffness signature of the tissue. It is anticipated that this stiffness signature could potentially be used to determine the health of the tissue and the possible presence of tumour/disease in the mesoscale.

The first step in the development of such a device is to demonstrate the feasibility of the sensor to characterise tissue properties. This is the focus of this thesis.

1.1 Motivation

By the age of 85, one in three Australian women and one in two Australian men will be diagnosed with cancer[4]. Early detection of cancer is an important factor in achieving favourable treatment outcomes[6]. Cancer often manifests itself as a tumour, which is defined as an abnormal mass of cells or tissue. The mechanical properties of the tissue (stiffness properties) are inevitably changed by the presence of a tumour[7]. Although not the only medical situation for which the characterisation of the mechanical properties of tissue could be useful, cancer has touched the lives of so many people that it provides a compelling case. Cancer does not discriminate; therefore cancer detection has proved to be a major motivation for this project. Further to this, even though human tissue has a wide range of mechanical properties, significant changes have been seen in cancerous
tissue [1] and as a result the mechanical properties of tissue have the potential for use as a diagnostic tool [8].

Cancer is characterised by a change in cell behaviour [9]. The first step in the development of cancer is initiation [10]. This is where cells become abnormal. Changes to cells are called mutations. Mutations can occur spontaneously, or as a result of exposure to carcinogens such as toxins and UV light, or due to genetic factors [9]. Normal cells require a number of mutations before they will turn into cancerous cells. Any cell in the body can become cancerous. Cancerous cells behave differently to healthy cells as they never stop dividing (they mature differently), they disobey normal cell signals, they can spread to other parts of the body and they stick together [11]. These characteristics give cancer cells the ability to rapidly multiply and develop tumours. Tumours cause changes to the mechanical properties of tissue compared to healthy tissue. These changes in tissue stiffness properties can be due to a number of factors including increased cell population, changed blood supply and presence of different cell types [11].

When cancer is first diagnosed it is characterised [12]. This characterisation does not change once assigned, no matter what the future progression of the cancer is. Characterisation of cancer is used to help plan patient treatment and predict patient outcomes. Cancer characterisation is a complex process that is dependent not only on the size and spread of the cancer, but also on the type of cancer and the amount of mutation seen in the cells. There are two common cancer characterisation systems: TNM [13] and cancer stages [6]. TNM stands for tumour, nodes and metastasis. Each variable is then assigned a number depending of its severity [6]. Figure 1.1 outlines the general rules for cancer characterisation using this method.

| T: Tumour: rated between 0 and 4 depending on the size and spread of the tumour within the tissue, with 4 having the greatest size/spread. |
| N: Characterises the amount of nodal involvement, rated between 0 and 3, with 0 having no nodal involvement and 3 having significant nodal involvement. |
| M: Metastasis - spread of cancer to other parts of the body. Given a 0 if there is no spread and a 1 if there is. |

Figure 1.1: Explanation of the TNM cancer characterisation method.

The cancer stages method is perhaps more commonly known. In this method there are commonly four stages of cancer. The basic criteria for these stages are outlined in Figure 1.2. If cancer is detected in its early stages there are generally more treatment options available for the patient, typically resulting in more favourable outcomes [6]. There
is potential in the future for the dynamic mechanical properties could be correlated to cancer characterisation systems.

Tumours are a good example of the significance of the scale when measuring the changes in the dynamic mechanical properties of tissue. A single cell would fall in the microscale typically on the order of ten micrometres, but depends on cell type. Cells can spontaneously change their stiffness properties with no significant long term effect on the health of the tissue. Therefore measuring changes in the dynamic mechanical properties at the microscale may lead to false negative diagnosis of tumours, resulting in unnecessary stress on the patient and on the healthcare system. In general, tumours tend to grow in size as they develop. Large tumours would fall in the macroscale (typically larger than 5 mm). Diagnosis at this stage is not commonly associated with good patient outcomes. It is therefore logical that in order to achieve the best outcome, tumours should be diagnosed in the mesoscale (0.1-1 mm). Characterising the dynamic mechanical properties in the mesoscale would allow this to happen.

| Stage 1: | Tumour is relatively small and contained to the tissue it originated from. |
| Stage 2: | The size of the tumour has increased from stage 1 however it has not spread to neighbouring tissue (for some cancer types: stage 2 can involve some nodal involvement). |
| Stage 3: | The tumour has further increased in size and has spread to surrounding tissue and the lymph nodes. |
| Stage 4: | The cancer has spread or metastasised to other parts of the body. |

Figure 1.2: Outline of the stages of cancer as per the cancer stages characterisation method.

1.1.1 Viscoelastic Materials

Human tissue is a viscoelastic material; this means that tissue has properties of both viscous and elastic materials. When a viscous material, such as honey is subject to an applied force it will gradually deform and stay deformed, whereas an elastic material, like rubber, will initially deform, but then return to its original state, once the applied force is removed. Viscoelastic materials display a combination of both these material properties. The level at which the viscous/elastic properties play a role in the overall stiffness properties of the material (such as human soft tissue tissue) depends on the rate or frequency at which it is tested. This is often referred to as the frequency dependence of tissue; or the dynamic properties of tissue (similarly for other viscoelastic materials)\[14\].
The stiffness of tissue can be characterised using the complex Young’s modulus \( E' \). The complex Young’s modulus \( E' \) is similar to the Young’s modulus \( E \), which is defined as the ratio of stress to strain, but takes into consideration both the elastic \( E' \) and viscous \( E'' \) components as shown in the next section. \( E' \) is often referred to as the real component or the storage modulus. Similarly \( E'' \) can be referred to as the imaginary component or the loss modulus. If the tissue were thought of as an object being dropped (e.g. a ball), the applied force could be considered to be the force exerted on the ball when it hits the ground. At the point of contact, some energy is dissipated by friction. The potential energy conserved allows the ball to bounce back to a new height below the height from which it was dropped. The storage modulus \( E' \) would relate to the height the object bounces back off the ground (the conserved energy) and the loss modulus \( E'' \) is related to the difference between the height the object bounced back and the height it was dropped (the energy dissipated through friction). This is graphically depicted in Figure 1.3.

Viscoelastic materials also have the added complication of being dependent on the measuring technique and preload. Colley et al. for example measured the elastic modulus of a viscoelastic material using two different methods [15], and found the results to be an

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Figure 1.3: Graphical depiction of an example of the combination of viscous and elastic characteristics within a viscoelastic material.
order of magnitude different. This was confirmed by Chatelin et al., who tested the mechanical properties of liver tissue and concluded that data on the mechanical properties of liver tissue is highly variable as they depend on the testing conditions and method [16]. Another group [17] tested the same material and were only able to replicate the results at a specific preload. They concluded that the complex Young’s modulus ($E'$) of viscoelastic materials is method and preload dependent.

The variations in reported dynamic mechanical properties of viscoelastic materials have hindered measurement, reporting and adoption of dynamic mechanical properties of human tissue as a clinical tool, as there is uncertainty in the results [16, 18]. Oudry et al. has tested four different measurement techniques in order to compare results and try to establish a gold standard [18]. A viscoelastic tissue mimicking phantom was used to verify elasticity results obtained using quasi-static compression (ultrasound elastography), dynamic mechanical analysis (DMA), vibration controlled transient elastography (ultrasound elastography) and hyper-frequency viscoelastic spectroscopy (more in depth discussion on the measurement techniques can be found in Sections 1.3 - 1.5). When comparing the Young’s modulus and shear wave speed, they confirmed variations between the four measurement techniques. They concluded that the elastic properties of viscoelastic materials depend on the measurement technique, making it extremely difficult to determine a reference method and to assess the elastic properties [18]. Suggestions were made as to the root of these variations including; variations in test phantoms; different boundary conditions of the sample tested, based on the requirements of the testing system; variation of measurement depth and variation in the frequency and way the shear waves (or other forces) are applied to the sample and preload. However they were not convinced that any of these observations sufficiently explained that variations seen between measurement techniques. It is therefore important to understand the measurement techniques used in order to create protocols and measurement calibrations that allow an accurate determination of the dynamic mechanical properties of viscoelastic solids, including soft tissues [18, 19].

1.2 PROPERTIES OF VISCOELASTIC MATERIALS

Despite widespread acceptance that the mechanical properties of tissue change with disease state [1], the documentation of the mechanical properties of biological materials in the literature is relatively new. The recent interest in this area has been driven by three
main research interests: the desire to understand more about material properties and how they change, to provide the information required to develop biologically inspired materials and to understand the interaction between biological and engineered materials [20]. Through the measurement of the mechanical properties of biological materials, it has been found that the elastic moduli cover a broad range of values, ranging from 0.0001 to 100 GPa [20]. Biological materials can be divided into four categories: ceramics, including bone and teeth; polymers, including ligaments and tendons; elastomers, including skin, muscle, blood vessels and soft tissue; and cellular materials, including cancellous bone [21]. These categories help to reduce the range of moduli specified for biological materials. Of most interest to the current thesis are elastomers. Elastomers have a reported modulus range of 0.1 to 100 MPa [21], however the measurement of the recorded modulus is dependent on the measurement method [15] and there have been reports of moduli in the 1 - 100 kPa range [22].

A number of different materials have been used to mimic the mechanical properties of biological materials. In general synthetic elastomers have similar moduli when compared to natural elastomers [21]. Agar and gelatine based samples have been tested and shown to have similar modulus properties to human tissue [23, 24]. PDMS samples with different stiffness have been compared to bone, brain and muscle tissue [22]. Hydrogels are also commonly used as tissue phantoms. Hydrogels have been confirmed to have comparable stiffness properties to biological tissue [25], however it was noted that different testing modalities produce different results and therefore all tissue phantoms should be compared to natural tissue samples for each measurement method. There are also concerns about the stability of current tissue phantoms over time [25, 24]. Having a better understanding of the mechanical properties of natural tissue may help to overcome this issue and hence create better tissue phantoms.

A number of abnormalities including cancer, fluid retention, inflammation and fibrosis can cause changes in tissue mechanical properties [1, 7]. For many years medical professionals have been using manual palpation to diagnose breast and prostate cancers. This is based on the theory that the mechanical properties of the tissue change as the tissue becomes diseased. Greenleaf et al. acknowledged that there was limited information on the mechanical properties of unhealthy tissue, but suggested that the elastic moduli may differ from the surrounding tissue by as much as 90-fold [1]. A few sources have stated that tumours are stiffer than normal tissue [3, 26–28]. More recently Plodinec et al. found that although cancer causes changes in mechanical properties compared to healthy tissue, these changes are not limited to increased stiffness. Changes to tissue mechanical
properties due to cancer can either increase or decrease stiffness [29, 30]. Cirrhosis of the liver increases the elastic modulus while fluid filled cysts decrease the modulus [3]. Wang and Larin set about creating an overview of what the difference in the Young’s modulus of healthy and unhealthy tissue are [2]. They found that in human breast tissue there was a difference of 124 kPa between a benign and malignant lesion, a 6.5 kPa difference between normal and fibrotic liver tissue, a 3 kPa difference between healthy and sickle red blood cells and a 300 kPa difference between healthy and calcified aortic wall [2]. This study aimed to create a reference point for all of the reported differences in Young’s moduli of cells, tissues and organs caused by changes to health. It appears that an extensive literature survey was completed, but again limited information was found. Most of the studies they looked at involved static measurements rather than dynamic measurements, but nonetheless gave a positive sign, that the mechanical properties of tissue can be used as a diagnostic method. It is hoped that measuring the dynamic mechanical properties of tissue will provide further information about changes in tissue properties. More information on measurement of human tissues are discussed in Sections 1.4 and 1.5.

1.3 Dynamic Mechanical Analyser (DMA)

A dynamic mechanical analyser (DMA) is a lab testing device that can be used to measure the viscoelastic properties of materials. The sample is first preloaded with a small force. An oscillatory (sinusoidal) stress is then applied to the sample and the resulting strain is measured (alternatively one can apply a strain and measure the resulting stress). Figure 1.4 illustrates the general principle of DMA theory. The blue line represents the applied stress and the black line, the resulting strain. The phase difference (\(\phi\)), between the two lines and the amplitude of the response is used to determine the stiffness of the material.

If an oscillatory stress (or strain) is applied to a purely elastic material it will result in an oscillatory strain (or stress) waveform that is in phase with the stress (or strain). If the same stress (or strain) is applied to a purely viscous material the resulting strain (or stress) waveform will be 90º out of phase. Under the same conditions a viscoelastic material (which includes the materials tested as part of this thesis) will be out of phase by between 0 and 90º. This information can be used to determine the complex Young’s modulus (\(E^*\)) of the material through the following Equations:
1.3 Dynamic Mechanical Analyser (DMA)

Figure 1.4: Schematic representation of stress-strain curves measured in a DMA. The blue line represents the applied stress, while the black line corresponds to the resultant strain.
\[ \tan(\phi) = \frac{E''}{E'} \]  

(1.1)

\[ E^* = E' + iE''. \]  

(1.2)

where \( \phi \) is defined as the phase shift between the applied stress and resulting strain and \( E^* \) is defined by the relationship between stress (\( \sigma \)) and strain (\( \eta \)) when subjected to oscillatory (dynamic) conditions as given by:

\[ \sigma_{i,j} = E^*_{i,j,k,l} \eta_{k,l}. \]  

(1.3)

The complex Young’s modulus (\( E^*_{i,j,k,l} \)) is theoretically a fourth order tensor (mathematical representation of a physical quantity) which describes the relationship between the second order tensors, \( \sigma_{i,j} \) and \( \eta_{k,l} \). The indices \( i,j,k,l \) relate to the Cartesian coordinate system in which the spatial dependence of the stress and strain are defined. However symmetry can be used to simplify the number of independent variables, assuming that the material is isotropic at the point where the measurement is taken. One must be careful with this assumption as it implies that values recorded for materials with mixed stiffness would record an averaged result. Reducing the number of independent variables allows the complex Young’s modulus (\( E^* \)) to be treated as it is in Equation 1.2, i.e. not as a fourth-order tensor. The complex Young’s modulus (\( E^* \)) consists of two components: the real elastic modulus (\( E' \)) and the imaginary viscous modulus (\( E'' \)) [31]. Unlike the standard Young’s modulus, the complex Young’s modulus (\( E^* \)), is the modulus at a point in time and can be affected by the rate/frequency of the applied stress (or strain). Sample stiffness, which unlike the modulus of the material is dependent on the physical dimensions of the sample, is defined as the force applied to the sample divided by the amplitude of deformation.

**DMAs** have been used to quantify the dynamic response of a number of materials. Of most interest to this thesis are optical fibre and human tissue. For more information on optical fibre see Section 1.6 and Chapter 2. The dynamic response of optical fibre is important, as the mechanical response of the optical fibre needs to be taken into consideration when analysing the output of the sensor. Stefani et al. looked at the dynamic response of optical fibre in the context of optical fibre sensors in general [32]. He found that in the frequency range between 10-100 Hz there was a frequency independent flat response, indicating that for measurements taken in this range the frequency would have no effect on the output from the optical fibre sensor [32]. Due to the limitations of the testing
configuration, they were unable to get clear measurements outside of this range. This result is significant as this is a similar frequency range proposed for testing human tissue, although the lower frequencies need to be further investigated.

It appears that the dynamic properties of some human tissues (muscle and bone) were first tested with DMA in 1966 [33]. However there does not appear to have been much further work published in this field until recently. With growing interest in imaging the stiffness properties of tissue (elastography, see Section 1.4) there has been a corresponding need to quantify the stiffness of the imaged tissue. A few groups have looked to DMA (or similar methods) to measure these dynamic properties with the aim of using these values to quantify images [34–38, 16].

In the eye, tactile surgical evidence suggests that healthy lens and cataract lens have different stiffness properties and that there is also variability in lens stiffness with age [39, 40]. DMA has been used to confirm this perceived difference. It was found that the compliance of human lenses was strongly dependant on the age of the person, namely compliance of the human lens decreased with age by a factor of 1000 over a lifetime [39]. Heys et al. have reported that cataracts lenses are stiffer than healthy tissue [40]. They found that different types of cataract lenses have different stiffness; in general nuclear cataract lenses are stiffer than cortical cataract lenses. Interestingly they also found that the difference in stiffness properties between healthy and cataracts lenses depends on the age of the patient, strengthening the argument that the stiffness of lenses is both age and health related. Heys et al. found that cataract lenses in patients aged 40-50 were 46 times harder than healthy lenses, and by the age of 70-80, even though the cataracts lenses were stiffer than that of the younger age groups, the hardness of the healthy lens had increased so much that the cataract lens was only 2.5 times stiffer than the healthy tissue [40].

The dynamic mechanical properties of bone have been investigated using DMA [41, 42]. Initial testing of bone with DMA has mainly been to assess the appropriateness of the measurement technique to examine the viscoelastic nature of bone. The viscoelastic properties of bone can have a significant impact on fracture instances [42]. DMA was able to successfully measure bone properties making it an effective tool to study the viscoelastic properties of bone. The DMA has subsequently been used to measure the effect of changes in collagen content in bone [41]. The amount of collagen in bone changes with age, menopause and metabolic disease, however there was not found to be a significant change in the viscoelastic properties of bone depending on the collagen content [41].

The mechanical properties of liver tissue has been the most widely studied, so there is no surprise that DMA has been used to evaluate these properties [16, 37]. It was found
that cirrhotic liver tissue was four times stiffer than healthy tissue [37] and that there was a linear increase in tissue stiffness, from healthy liver tissue, for increased levels of fibrosis (cirrhosis is severe fibrosis). DMA has also been used to measure the mechanical properties of other human tissue including the uterus [35], cervix [43] and mitral valves [44].

This thesis aims to use the DMA theory to calculate the dynamic mechanical properties of viscoelastic solids using an optical fibre sensor and as a standard to validate results calculated using the optical fibre sensor.

1.4 Elastography

Elastography is an emerging medical imaging-based technique for looking at tissue stiffness properties. Elastography works by applying a force to the tissue and imaging the tissue’s response. The applied force deforms the tissue and an image is taken of the tissue’s response [45]. The force can be applied to the tissue in a variety of different ways, including an internal source such as the heart beat or an artificial external force. Elastography produces elastographs which are images of stiffness properties. Elastography is considered to be an important diagnostic tool as it reduces the need for invasive procedures including biopsies, provides the clinician with more information about tissue properties and the health of the tissues tested and it helps to further develop our understanding of tissue disease states [45]. Although most commonly used to diagnose changes in the liver [46, 47], elastography has also been used to detect changes in other human tissues, including breast [28, 48, 49, 30], bladder [50], brain [51], heart [52], skeletal muscle [45], pancreas [53, 54] and prostate tissue [55]. It is believed that in the future elastography will be used in a much broader range of applications. Elastography has been found to reduce the number of liver biopsies dramatically [56] and was found to be more specific and produce fewer false positive results in breast cancer screening compared to conventional ultrasound [49].

There are a number of different types of elastography available [57, 36], the most commonly reported are ultrasound elastography [58, 54, 3, 59–68], magnetic resonance elastography (MRE) [69, 70, 7, 45] and optical coherence elastography (OCE) [71–77, 70, 78–80]. Ultrasound elastography is the most commonly used mode of elastography to date. This is probably due to the ease of applying forces to tissue using an ultrasound probe. Ultrasound elastography can be divided into four main categories distinguished by the
way in which the force is applied to the tissue. The simplest is compression elastography. In this case the force is often applied manually, such as pushing the ultrasound probe against the tissue and includes the use of internal forces. Compression elastography has been used for the detection of breast, prostate and thyroid cancer, characterisation of intra-vascular plaque and tendonitis assessment\textsuperscript{57} as well as stiffness measurements of forearm and lower limb soft tissue\textsuperscript{81}. Sonoelastography is a form of ultrasound elastography where the force is applied to the tissue using ultrasound waves; most commonly shear waves\textsuperscript{82}. Sonoelastography has been mainly used in the detection of prostate and liver disease\textsuperscript{57}. Similar to sonoelastography, transient elastography uses propagating shear waves to apply a force onto the tissues being tested, but instead of a constant stream of waves this method uses short bursts of vibration\textsuperscript{83}. This is done to avoid interaction between propagating and reflected waves. Transient elastography has been extensively used to determine the mechanical properties of the liver\textsuperscript{46}, as well as detection of breast tumours and muscle stiffness\textsuperscript{57}. The fourth method is acoustic force radiation\textsuperscript{84}. Unlike the other methods mentioned, this method applies the force to the tissue via internal tissue excitation. This is achieved via focused ultrasound pulses\textsuperscript{57}. Acoustic force ultrasound elastography has been applied to breast, abdominal, heart and liver tissue\textsuperscript{57}. Although ultrasound elastography is widely used due to the relatively low cost and real time image acquisition, it is limited by poor spatial resolution (theoretical limit of 1.5 mm)\textsuperscript{57}.

Elastography using a magnetic resonance imaging (MRI) machine to image tissue response to an applied force, instead of ultrasound is known as magnetic resonance elastography (MRE)\textsuperscript{7}. Due to the expense and increased acquisition time, compared to ultrasound elastography, MRE has not featured in the literature as prominently. It does however have the advantage of providing increased resolution (35 µm) and field of view\textsuperscript{57}. As with ultrasound elastography, the force applied to the tissue during MRE can be through either an internal or external source\textsuperscript{82}. The most common method of force production is high frequency shear waves\textsuperscript{57}. MRE has been used in the characterisation of liver, breast, muscle, brain and lung tissue\textsuperscript{57, 82}.

The optical imaging technique, optical coherence tomography (OCT), can be used as an elastography method, and is referred to as optical coherence elastography (OCE)\textsuperscript{2}. OCE is favoured for its high resolution (5-10 µm), but is limited by a small field of view and limited tissue penetration\textsuperscript{57}. Researchers have tried to overcome this potential issue, by embedding an OCE probe into a needle that can be pushed into the tissue\textsuperscript{85}. OCE has
been used to measure changes in tissue properties in the cornea, soft tissue, artery wall, muscle, skin and individual cells [57, 2].

Elastography methods which rely on imaging modalities, as described above, can be expensive and bulky. They also rely on the availability of imaging equipment and trained imaging professionals. In addition although elastography is usually on the mesoscale, it often only provides a stiffness comparison at a single frequency instead of the desired quantitative values over a range of frequencies. This potentially limits the diagnostic value and means that the results will be highly dependent on the measuring technique [18]. Currently there appears to be a gap in knowledge in this regard. In order to better understand these images, the stiffness properties of tissue and how they change when they become diseased, there appears to be a need for a method of quantifying the stiffness properties of tissue in the mesoscale, in vivo. The closest elastography technique to this requirement is OCE. In a recent paper [72] OCE has been used to demonstrate a frequency dependent contrast between different stiffnesses of silicone phantoms. The paper looks at displacement amplitude and phase but stops short of relating these values to the complex Young’s modulus ($E^*$) i.e. this is not a fully quantitative method. Adie et al. were however able to measure the frequency dependent (0 - 1 kHz) differences between silicone phantoms (PDMS) with different stiffness [72]. They demonstrated that an increase in the measured power of the system was correlated with an increase in the stiffness of the sample tested. A feature that appears to be a resonance was also recorded in the system at approximately 122 Hz. A peak in the recorded signal can be seen at this point, for all the samples tested. The signal peak at the resonance frequency also increased with increasing sample stiffness [72]. This was thought to be due to stiffer materials favouring higher resonant frequencies. Images of a PDMS sample with a 1 mm inclusion where also successfully produced at different frequencies, providing images of relative stiffness. While OCE shows great promise, it requires the use of a relatively complex optical system, making the system potentially unstable and difficult to make portable.

1.5 OTHER TECHNIQUES FOR MEASURING DYNAMIC MECHANICAL PROPERTIES

There are a few other laboratory-based techniques that can be used to determine the dynamic stiffness properties of tissue. These include, but are not limited to, nano indentation [86, 87] and the atomic force microscope (AFM) [88]. These techniques are quantitative; however measurements are not taken on the measurement scale of interest in this
thesis. These techniques operate on the microscale or nanoscale. This scale is useful in research at the cellular level, however is usually confined to the laboratory environment. These techniques are also often limited to a single frequency. Further to this, even though these techniques are used regularly in the laboratory environment, their application to soft tissue is limited [18].

Indentation as a method of measuring the mechanical properties of viscoelastic materials, including biomaterials, can be achieved on both the microscale (microindentation) and the nanoscale (nanoindentation). The indentation principal uses the relationship between indenter force and penetration depth during loading and unloading to determine mechanical properties, most commonly elastic modulus, shear modulus and hardness [86, 89]. Indentation is a popular laboratory technique for measuring mechanical properties, as limited sample preparation is required and the testing procedure is usually automated and simple to use. Indentation relies on the use of an indenter to indent (and often penetrate) the sample. Indenters can come in a number of shapes with cylindrical, conical and spherical probes being most common [86]. The shape of the indenter dictates the relationship between contact load, penetration depth and the mechanical properties of the material tested, due to the changes in contact stiffness and area. Indentation has been used to measure the mechanical properties of human tissue, including meniscus, cartilage, tooth enamel and skeletal muscle in the laboratory environment [90]. Indentation has also been used to measure the mechanical properties of manufactured biomaterials, designed to mimic natural biomaterials in order to try and create new materials that have improved similarity to the materials they are mimicking, including hydrogels [90].

There are two approaches to measuring the mechanical properties of biomaterials using AFM: the traditional indentation force/distance method, which gives you access to modulus recordings over a very wide range of elasticities, but no spatial information [91] and the more recently developed nanoscale force mapping techniques, Quantitative Nanomechanical Mapping-AFM.

The indentation AFM method is an extension of the indentation method mentioned above, using the AFM tip as the indenter. The indentation AFM technique has been used to measure the mechanical properties of microtubules, filaments, collagen, fibrils and proteins [91], as well as cancer cells [92–94], including breast [29, 95], liver [96] and kidney cancer cells [97].

Each brand of AFM has slightly different technologies, but probably the most developed (certainly the most commonly used) is the PeakForce mode from Bruker [98]. The
AFM force mapping method uses the relative movement between the AFM probe and the sample to measure the mechanical properties of the sample. Using the PeakForce mode from Bruker, the AFM not only moves relative to the sample, but intermittently makes contact with the sample, recording the contact force \[98\]. PeakForce AFM has been used to measure the mechanical properties of polymers, viruses including herpes, cellulose \[98\], bacteria and cells \[99, 100\].

1.6 Optical Fibre Sensors

The technique proposed for determining dynamic mechanical properties in this thesis relies on an optical fibre sensor. Optical fibre is a thin light guiding structure typically made from silica glass \[101\]. Common single mode optical fibre has three layers. The innermost light guiding layer is the glass core, which is surrounded by the glass cladding and an outer protective acrylic coating as shown in Figure 1.5. Light is guided in the core of the optical fibre by total internal reflection. This means in order for light to pass through the core of the optical fibre it reflects off the boundary between the core and cladding. Light hits the boundary and is subject to total internal reflection.

![Figure 1.5: Representation of single mode optical fibre showing the glass core and cladding covered by a protective polymer coating. Please note this image is not to scale.](image)

Optical fibre sensors can be used to sense a variety of parameters such as strain or force, temperature and bending \[5\]. A common type of optical fibre sensor is a fibre Bragg grating (FBG) \[101\]. FBGs consist of periodic changes in the refractive index of the core of an optical fibre; please refer to Figure 1.6. When light passes through the optical
Figure 1.6: Visual description of how an FBG works. The incident light passes through the optical fibre, interacting with the FBG. Light of a specific wavelength (Bragg wavelength) is reflected. All other wavelengths of light are transmitted.
fibre, it interacts with the refractive index changes, and light of a specific wavelength 
(the Bragg wavelength) is reflected and all other wavelengths of light are transmitted. FBG sensors are known to have good repeatability and sensitivity $^{[102]}$. They boast long failure times (>1 million cycles), fast response time (<1 μs) and excellent thermal stability at temperatures below 3000 °C $^{[103]}$.

1.6.1 Optical Fibre Sensors in Medical Applications

Optical fibre sensors are currently used in the medical field for the sensing of a number of different parameters, due to their desirable properties, including small size and weight, biocompatibility, multiplexing capability and low susceptibility to electromagnetic radiation. Optical fibre sensors have been used for both monitoring and diagnostic applications. The types of optical fibre sensors used in medical applications fall largely into three main categories: 1) chemical sensors; 2) immunoassay sensors; and 3) physical sensors $^{[104, 105]}$.

1.6.2 Optical Fibre Chemical Sensors

Optical fibre chemical sensors have been used to detect the presence of different substances including vapours, glucose, oxygen concentration $^{[106–108]}$, protein, drugs and drug diffusion $^{[109]}$, pH and pesticides $^{[105]}$, within the medical environment.

Eytan et al. used IR-spectroscopy via an optical fibre to perform blood tests $^{[110]}$. They were able to successfully measure the concentration of protein, cholesterol, urea, uric acid and creatine in blood samples with good correlation to standard pathology tests. The optical fibre method provided the benefit of real-time analysis instead of sending the blood to a pathology laboratory, as well as reducing the need for reagents or other additives to be added to the blood.

Stitzel et al. used optical fibre chemical sensors when designing and building an artificial nose $^{[111]}$. Colorimetric and fluorescence-based optical fibre sensors were used to detect small amounts of vapour in the air and correlate these to different smells. Cross-reactive sensor arrays combined with pattern recognition algorithms were used to distinguish between different odours. There have also been reports in the media that “smelly” volatile molecules could be used to diagnose diseases, including cancer.
1.6.3 Optical Fibre Sensors for Immunoassays

Immunoassay is the process of measuring or detecting specific substances through their properties as antigens or antibodies. With regards to optical fibre sensors, this is most commonly achieved using spectroscopy or fluorescence i.e. detecting changes in the optical properties of the substances being tested[112–114].

Eltzov et al. explored the use of biosensors for molecular diagnostics, including pesticides, air toxicity, viruses, hormones and drugs[115]. They found many advantages with using optical fibre, including size, flexibility, low cost of production and chemical inertness.

Optical spectroscopy has also been used in the medical field to diagnose gastritis[116], detect cancer and other tissue pathologies using a Fabry-Perot interferometer to measure changes in photoacoustic and photothermal tissue responses[117], detect changes in skin[118] and tumour diagnosis[119] using IR-spectroscopy, as different disease states of tissue have individual patterns.

1.6.4 Optical Fibre Sensors for Physical Quantities

Optical fibre sensors used as physical sensors appear to be the most commonly applied in medical applications. Optical fibre sensors are often used to monitor temperature, displacement, force and pressure within the medical field[120]. More specifically, FBG sensors have been used to measure bone strain, pressure stress and strain magnitudes of orthopaedic joints, foot pressure, pressure distribution of intervertebral discs, dental biomechanics, chest wall deformation, human machine interfaces, pressure and shear stresses in amputee sockets, pressure areas for vulnerable patients to prevent pressure sores and human body kinematics[121].

Fibre optic thermometry sensors have been used to track the breathing cycle of patients by measuring the temperature difference between the air being expired and inhaled[122]. Optical fibre FBG sensors have been used to measure the heat resistance of cells[123], the temperature of soft tissue[124] and for temperature monitoring within intelligent clothing[125]. Healthy cells have different heat resistance compared to cancer cells. Therefore measuring temperature changes could be used to diagnose tissue health as well as for monitoring thermal treatments.
Optical fibre displacement sensors have been used to measure the angular displacement of joints [126]. Optical fibre accelerometers are also commonly available for healthcare applications. Optical fibre tactile sensors are also used to assist physicians during minimally invasive surgery [127–129]. Tactile sensors allow surgeons to “feel” tissues via haptic feedback, in place of direct contact with the tissue. To date these sensors are manually driven (not driven over a frequency range) and do not offer quantitative analyses, but rather real-time force feedback.

An FBG sensor has also been used to measure the forces incident on the delicate structure of the inner ear during the insertion of cochlear electrode arrays [130]. Forces exerted on the cochlea during the insertion of cochlear electrode arrays can damage the cochlea leading to reduced success of the cochlear implant [131]. The FBG sensor developed by Wade et al. allowed for the insertion forces to be measure in real time, helping surgeons to reduce unintentional forces and potentially reduce damage to the cochlea [130].

Optical fibre plethysmography has also been used to measure torso displacement during breathing [132] and displacement within the myocardial walls, allowing the measurement of heart chamber volume [133, 134]. There are also reports of optical fibre sensors being used in dentistry to measure displacement [135].

The most common optical fibre pressure monitor is the intracranial pressure monitor. Intracranial pressure is an important measure in patients with head injury and neurological patients. Although many studies have shown that optical fibre intracranial pressure monitors are safe and reliable [136–142], they do not appear to be widely used. This may be due to physicians being unwilling to move away from the ‘gold standard’ external ventricular drain. The advantage of this type of monitoring is that it allows drainage of excess fluid; however optical fibre sensors have the advantage of being easier to place, and are less susceptible to infection and haemorrhaging [138]. On the other hand, current optical fibre sensors cannot be calibrated in vivo. This issue can easily be overcome by increasing the stability of the sensors. It is hoped that in the future optical fibre sensors will be used more frequently as intracranial pressure sensors in patients that do not require ventricular drainage.

Other types of optical fibre pressure sensors include an FBG sensor to measure grip strength [143], ballistocardiography sensors [144, 145], Fabry-Perot sensors to measure fluid pressure transduction [146] and intramuscular pressure sensors [147], including an FBG sensor to assess pelvic floor function [148].

Variation in the mechanical properties of embryonic tissue is correlated to birth defects. Due to the delicate nature of embryonic tissue, measuring these changes without damag-
ing the tissue can be difficult. Moore used an optical fibre system to measure the dynamic mechanical properties of embryonic tissue [149]. The approach recognises the importance of measuring the dynamic mechanical properties of viscoelastic material, but only measures percentage strain, stopping short of calculating the complex Young’s modulus ($E^*$).

The approach used a cantilevered optical fibre force sensor to measure probe tip position and deflection, correlating it to tissue deformation and force respectively. The design contains a complicated actuator structure that seems impractical for in vivo use, but good for baseline data of tissue because it allows measurement of stress/strain in different directions/axes. The system proposed is very elaborate, possibly making portability difficult, but successfully demonstrated the measurement of the dynamic mechanical properties of embryonic tissue, providing some insight into the tissue’s response to an applied shear stress. The results presented to date are limited as the research focuses on the measurement technique. Although initial results are presented in vitro and it is concluded that more work is required to optimise the system, perhaps further highlighting the difficulty in measuring the dynamic mechanical response of human tissue.

Optical fibre FBG sensors have proven popular for medical applications, mainly due to their small size and weight, stability and immunity to electrostatic, electromagnetic and radio frequency interference. An FBG sensor is the type of optical fibre sensor used in this thesis to try and determine the dynamic mechanical properties of viscoelastic solids.

### 1.7 FBG Sensing and Fabrication

FBGs are fabricated by writing refractive index changes (vertical lines) into the core of the optical fibre (commonly using a laser). The distance between the refractive index changes, known as the FBG period, is generally constant. The FBG period can be changed for example by applying a force to or changing the temperature of the fibre, resulting in a shift in the reflected wavelength. This wavelength shift can be measured using a number of methods which will be discussed later. Linear shifts in Bragg wavelength have been observed when strains of up to 1% [150] and compressions of up to 2.7% [151] were applied. The wavelength of the reflected light, $\lambda_B$, can be determined by:

$$\lambda_B = 2n_{eff}\Lambda,$$

where $n_{eff}$ is the effective refractive index of the propagating mode within the fibre at the Bragg wavelength and $\Lambda$ is the grating spacing, as defined in Figure 1.6 [101].
The wavelength shift $\Delta \lambda_{BS}$, for an applied strain $\Delta \varepsilon$ can be determined by:

$$\Delta \lambda_{BS} = \lambda_B (1 - \rho) \Delta \varepsilon,$$  \hfill (1.5)

where $\rho$ is the photoelastic coefficient of the fibre [101]. The temperature change $\Delta T$, related to a wavelength shift $\Delta \lambda_{BT}$ is given by:

$$\Delta \lambda_{BT} = \lambda_B (\alpha + \xi) \Delta T,$$  \hfill (1.6)

where $\alpha$ is the thermal expansion coefficient for the optical fibre and $\xi$ is the thermo-optic coefficient of the optical fibre [101].

There are two common fabrication techniques for FBGs: the two beam interferometer method and the phase mask PM method [103].

The two beam interferometer relies on the interaction between two beams of light, usually from a laser and beam splitter, to produce an interference pattern within the core of the optical fibre, hence creating an FBG through photochemical modifications of the glass. There are a number of different two beam interferometer FBG fabrication methods, including the holographic method and the source-tuneable interferometer method [152, 103]. The two beam interferometer method is the traditional FBG fabrication method [103], as such it has been well documented in the literature. The main characteristic of the two beam interferometer method is that the method used determines how the Bragg wavelength of the manufactured FBG can be selected. The Bragg wavelength can be selected in the holographic method by changing the incident angle between the two beams and the optical fibre [103]. The source-tuneable interferometer method is perhaps more simple as it allows for the Bragg wavelength to be selected by tuning the wavelength of the laser used to produce the two light beams [103].

The PM technique uses a PM to photo-imprint an interference pattern on the optical fibre. A PM is a diffractive optical element that can be used to create a lateral interference pattern [103]. It is this interference pattern that determines the Bragg grating pitch ($\Lambda$):

$$\Lambda = \frac{\Lambda_{pm}}{2},$$  \hfill (1.7)

where $\Lambda_{pm}$ is the grating period of the PM. In order to fabricate an FBG, the interference pattern formed by the PM is photo-imprinted into the core of the optical fibre as a refractive index modulation (grating) using a laser (commonly ultraviolet (UV)). A diagram of the PM fabrication technique is shown in Figure 1.7;

The PM and a stripped (outer coating removed, see Section 2.3) section of optical fibre are held parallel to each other and approximately one fibre diameter apart. Light from
Figure 1.7: Schematic of the phase mask (PM) FBG fabrication technique (not to scale).
the UV laser is incident on the PM through a cylindrical lens perpendicular to the PM. The laser is then scanned along the PM at a set speed for the length of the required FBG. The length of the FBG can be varied by controlling the scanning distance of the laser. The FBG length is limited by the length of the PM (upper limit) and the diameter of the laser beam (lower limit). The length of the FBG, the scanning speed, the strain at which the fibre is held and the PM (period and quality) all contribute to the bandwidth and Bragg wavelength of the FBG.

Gratings with wider bandwidths can be written using a chirped PM. The fabrication technique is the same, but the interference pattern on the PM is chirped, usually with a linear change in the grating period (\( \Lambda \)), as a function of position along the PM. These are known as chirped FBGs, which are commonly used as filters.

The fabrication of an FBG is dependent on the photosensitivity of the fibre core. Good photosensitivity allows the refractive index of the optical fibre core to be modified with minimal changes to the structure of the fibre. In order to increase the sensitivity of the fibre, a high-pressure chamber is often used to diffuse hydrogen (H\(_2\)) molecules into the fibre. This allows the UV laser light to generate hydroxyl groups in the glass core of the fibre, which lead to large permanent index changes. Using this method FBG reflectivity often approaches 100%.

In comparison with the two beam interferometer method, the PM technique has a number of advantages [103]: (1) the Bragg wavelength of the FBG is defined by the pitch of the phase mask, not the wavelength of the laser; (2) the phase masks are computer generated, offering good repeatability and the prospect of mass production; (3) the method only requires a single laser beam, adding to the stability of the set up; (4) the laser used can be less coherent, reducing the costs of the set up. This thesis relied on the PM method to produce the FBGs used in the project.

1.8 SUMMARY

This project aimed to take the current understanding of DMA, elastography and FBG sensors and build on them in order to develop a novel method to measure dynamic mechanical properties of a viscoelastic solid, namely PDMS, in the mesoscale. It is hoped that the technique could be expanded in the future to create a device that could aid in the early detection of poor tissue health in the mesoscale and help to improve the treatment outcomes for patients.
This thesis contains four subsequent chapters: the following chapter looks at the manufacture, calibration and mechanical properties of optical fibres, focusing on deflection and buckling force (Chapter 2). Chapter 3 explores using a DMA to test the dynamic mechanical properties of PDMS (a viscoelastic tissue phantom material) and optical fibre. Chapter 4 covers the testing of the FBG sensor to measure the complex modulus of PDMS with different stiffness, during bench top analysis. Chapter 5 focuses on moving the novel approach towards a diagnostic medical device to determine changes in dynamic tissue properties. The main findings of the thesis and recommendations for future work are discussed in the final chapter (Chapter 6).
2

CALIBRATION AND MECHANICAL TESTING OF OPTICAL FIBRE SENSORS

2.1 INTRODUCTION

The sensor used in this thesis is a type of optical fibre sensor. Optical fibre sensors generally rely on light propagating down the core of the optical fibre. When an optical fibre is bent or buckled the light propagation characteristics may change and some of the light may be lost. Therefore mechanical testing of the sensors is important. It gives information about the maximum force that can be applied to the sensor before it fails, or the strain transfer changes. Mohammad et al. [150] acknowledged that buckling limits the range of forces that can be measured using FBGs. Once the sensor has failed (buckled or bent) the sensor reading can no longer be trusted as it is difficult to tell how much light has been lost. This is due to the intensity and the wavelength of the reflected light being affected, potentially invalidating the output of the FBG interrogation system (see Section 1.6). The design, manufacture and calibration of the FBG sensor must take these limitations into account in order to allow it to be used as a complex modulus sensor. These issues are further discussed in this chapter.

The mechanical testing was performed in two parts: deflection force and buckling force. Deflection force is described as the force required to deflect the sensor when the force is applied perpendicular to the longitudinal axis of the sensor. Buckling force is the force along the longitudinal axis required to buckle the sensor. Mechanical testing was performed on optical fibre with three different diameters: 50 μm, 80 μm and 125 μm. Both mechanical testing experimental designs were based on the experimental procedure used by Patrick and MacFarlane [153] and Resbscher et al. [154] to test the mechanical properties of cochlear electrode arrays. Cochlear electrode arrays have a similar long tubular shape to the optical fibre sensor, which justifies the adoption of these methods.
The buckling force was also tested using a piece of optical fibre containing an FBG, instead of a plain optical fibre, to look at the output of the FBG during buckling. It is important to understand the deflection and buckling behaviour of optical fibre mechanical sensors so that buckling or deflection can be avoided or accounted for.

2.2 FBG SENSING SYSTEM

The FBG sensing system is comprised of not only the FBG, but also the hardware used to interrogate the FBG signal. Interrogation of the signal is pivotal in determining the wavelength shift of the FBG. An example of the spectrum of an FBG manufactured for use in this thesis is shown in Figure 2.1. For the present application, the interrogation system should provide adequate resolution and be cost effective [103]. There is ongoing research interest in the development of interrogation schemes. Some of the interrogation schemes reported include bulk optic spectrometers, tuneable filters, edge filters, interferometric techniques and the use of swept wavelength laser sources [103, 155].

The FBG sensor system used in this thesis takes advantage of the matched FBG interrogation scheme. The matched interrogation system relies on the pairing or matching of the output of two FBGs. As shown in Figure 2.2, the matched FBG interrogation system consists of a sensor FBG and a reference FBG that are matched so their spectra overlap. As a result, the reference FBG acts as a matched filter, with a transmission that is dependent on the precise wavelength reflected from the sensor FBG. This results in a voltage output that is dependent on the amount of overlap of the two spectra, as shown in Figure 2.3. Optimising the matching or overlap of the sensor FBG and the reference FBG improves the sensitivity of the system. Similarly the system can be further optimised by choosing a reference FBG and a sensor FBG that are well matched. Grating specification, including Bragg wavelength and full width half maximum (FWHM), determine how well matched the FBGs are. These FBG parameters can be selected during the manufacture process by changing the writing parameters including but not limited to the PM used, the grating length and the strain applied to the optical fibre. The writing parameters used to make the sensor FBG used in this thesis are listed in Figure 2.1. The matched FBG interrogation scheme supports all of the desired characteristics of an interrogation system while also being relatively simple in configuration, robust (no moving parts), compact, lightweight, and can be designed for low power consumption [155]. These characteristics are important design specifications for affordable portable devices.
Figure 2.1: Spectrum of FBG sensor used to measure the dynamic mechanical properties of tissue. The writing parameters used to produce this FBG were: PM grating period 1.0768 µm, grating length 2 mm, speed 0.15 mm/min and strain of 135 g. The grating parameters are discussed in Section 2.8.2.
Figure 2.2: Simplified schematic of the matched FBG interrogation system used as part of the FBG sensor system.
As mentioned in Section 1.6, when the sensor FBG is subjected to a change in the measurand (i.e. force) there is a resultant wavelength shift. The shift in the reflected wavelength changes the amount of overlap of the matched FBGs. Changes in the matching of the FBGs results in a shift in the output power of the system, as shown in Figure 2.3. This change in power can be calibrated to determine the change in the measurand. A second detector and a coupler are added to the system to correct for changes in the output power resulting from fluctuations in the intensity of the transmitted signal or light source. A third FBG (chirped FBG) is used to filter the light source incident on the reference and sensor FBGs. This helps to increase the sensitivity of the system by decreasing the amount of light at wavelengths not relevant to the required measurement range of the system. The spectrum of the chirped FBG used in the interrogation system is shown in Figure 2.4.

Figure 2.3: Matched FBG output, showing the effect of different levels of spectral matching between the sensor FBG (FBG 1) and the reference FBG (FBG 2). Image adapted from [155].
Figure 2.4: Spectrum of chirped FBG used in the FBG interrogation system as a light source filter. The writing parameters used to produce this FBG were: PM grating period 1066.5 µm with a 13.16 nm/cm strain rate, grating length 0.8 mm, speed 0.1 mm/min and strain of 80 g.
Compared to the system of \[155\], the current system also includes a chirped FBG (light source filter FBG), which is used to filter the superluminescent LED as shown in Figure 2.5. Using a broad band light source allows the system to be more flexible, i.e. FBGs can be used with different Bragg wavelengths and the intensity of the reflected light is kept constant as the wavelength shifts, this is not possible with a laser. However, the superluminescent LED is broader than required for a particular pair of sensor and reference FBGs, resulting in excess detector noise due to stray light. The light source filter FBG filters out the unwanted wavelengths of light, converting the broadband light source into an approximately “top hat” profile light source, thus helping to improve the signal to noise ratio. After reflecting from the chirped FBG, the light is passed through a circulator to the sensor FBG. This is the sensor used to measure changes in the measurand. The reflected light from the sensor grating returns to the circulator and is passed through to the reference FBG via a 50:50 coupler. This FBG is temperature controlled (not preloaded) and static, acting as a constant comparison point for the sensor grating and allowing for control of the temperature difference between the two FBGs. This allows the amount of matching between the spectra to be set as FBGs are sensitive to temperature changes. The temperature of the reference FBG will in part determine the reflected wavelength of the FBG and in turn the amount of matching between the reference and sensor FBGs. Controlling the temperature difference between the two FBGs allows measurements to be taken with the greatest sensitivity or the greatest range. The greatest sensitivity of the system is achieved when the matching between the reference and sensor FBGs is optimised, allowing for smaller forces to be detected. After filtering by the reference FBG, the light passes to a detector. The remainder of the light from the coupler passes to a second detector. The output of the system is the ratio of the two detectors, \( \frac{V_1}{V_2} \). This helps the system to better account for changes in temperature and input light intensity, thus increasing the accuracy of the system.

FBGs are sensitive to both strain and temperature, as shown by Equations 1.5 and 1.6. The effect of temperature fluctuations can be reduced by having the reference and sensor FBGs in close proximity to each other \[155\]. If using the sensor to detect changes of tissue properties \textit{in vivo} this may not be possible. Fortunately the human body has a very stable internal temperature. In this situation it is important to stabilise the temperature of the reference FBG. This is achieved through the control of a Peltier heater device, as shown in Figure 2.5.

It is important to make sure that the temperature of the reference FBG is as consistent as possible, as small fluctuations in temperature can affect the matching of the reference
Figure 2.5: Block diagram of FBG sensor system, based on the match FBG interrogation system.
and sensor FBG and hence the force measured. The relationship between the apparent changes in force, $\Delta F$ due to a change in temperature, $\Delta T$ can be calculated using the FBG equations presented in Section 1.7. By setting $\Delta \lambda_{BS}$ to equal $\Delta \lambda_{BT}$ in Equations 1.5 and 1.6 it was determined that:

$$(1 - \rho) \Delta \varepsilon = (\alpha + \xi) \Delta T,$$

substituting $\rho = 0.2$ ([101]), $\alpha = 0.55 \times 10^{-6}$ and $\xi = 8.6 \times 10^{-6}$ [101] allows for the above equation to be further simplified to:

$$\Delta \varepsilon = (1.2 \times 10^{-5}) \Delta T.$$  (2.1)

The relationship between the force ($\Delta F$) and strain ($\Delta \varepsilon$) is:

$$\Delta \varepsilon = \frac{\Delta F}{(\pi r^2) E},$$  (2.3)

where $r$ is 62.5 $\mu$m and $E$ is equal to 72.5 GPa [150].

Therefore

$$\Delta F = \frac{\Delta T}{100} \ [N].$$  (2.4)

Measurements of the temperature of the block used to control the temperature of the reference FBG in this system were taken using a scientific thermometer (Tesco, 735). The results of the temperature measurements are shown in Figure 2.6 (over a few hours) and Figure 2.7 (over a few minutes). As can be seen in Figure 2.6 there is a periodic 0.5 °C change in temperature over an hour, this is equivalent to a 5 mN force difference. In Figure 2.7 we see a temperature difference of about 0.1 °C, which equates to a force change of 1 mN. This instability will need to be considered when using the FBG sensor system. Theoretically a more stable temperature control system could be used to help improve accuracy.

The linearity and sensitivity of the system can be enhanced by optimising the wavelength difference between the sensor and the reference FBG and by determining the range of wavelength shifts for which the system is most sensitive. The wavelength difference that results in the greatest sensitivity and the range of sensitive wavelength shifts depend on the temperature difference and the FWHM of the gratings used [155].
Figure 2.6: Results of temperature measurement of the reference FBG over a period of a few hours, using a scientific thermometer (Tesco, 735).

Figure 2.7: Results of temperature measurement of the reference FBG over a period of a few minutes, using a scientific thermometer (Tesco, 735).
2.3 PREPARING OPTICAL FIBRE FOR TESTING

The testing of optical fibre mechanical properties was completed on both plain optical fibre and optical fibre containing an FBG. Making individual FBGs with specific Bragg wavelengths can be time consuming and they can be easily damaged in the laboratory research environment. As a result, it was decided that most of the mechanical testing would be completed with plain optical fibre. The plain optical fibre was expected to have similar mechanical properties to the optical fibre containing the FBG, as it is the same optical fibre, without the photo imprinting. This allowed for the testing to be completed in a timelier manner, and left all of the FBGs produced to be available for sensing purposes instead of risking damage during testing.

In order to prepare plain optical fibre for mechanical testing, the fibre was cleaved at the tip, at 90°. To achieve this, about 15 mm of the protective acrylate coating was removed from the tip of the fibre, using a mechanical stripping tool for the 125 μm (Fibercore, SM1500) and 80 μm (Newport Corporation, F-SBD) diameter fibres and paint stripper for the 50 μm (Corning, SMF-28) diameter fibre. The newly stripped optical fibre was then cleaned with ethanol. A Fitel S323 optical fibre cleaver (125 μm) or York FkII Fibre cleaver (80 μm and 50 μm) was used to cleave the fibre leaving a length of approximately 10 mm at the tip of the fibre without the protective coating. These dimensions are comparable to those used for the FBG sensors, as discussed below.

2.3.1 FBG Sensor Development

The FBGs were written in two different types of hydrogen (H₂) loaded single mode optical fibre with 244 nm radiation from a frequency doubled argon-ion laser (Coherent SabreFred ect) using the scanning PM technique, see Section 1.6. The optical fibres used were Corning, SMF-28 (125 μm) and Newport Corporation, F-SBD (80 μm). The gratings are written into the core of the optical fibre in the region where it has been mechanically stripped and cleaned with ethanol; see above section (Section 2.3). The centre wavelength of the gratings was approximately 1560 nm. The spectral properties of the FBG sensors were characterised by measurement at room temperature and zero strain using a swept wavelength system with 3 pm resolution (JDS Uniphase SWS15100). The details of the writing parameters used to produce the sensor FBG are listed in Figure 2.1. As the FBG sensors were produced individually there is some variability between the gratings (δλₜₐₜₚₐₚₚₜₚₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑₚₑ₢
approximately 0.13 pm for the gratings used in this work). This variability can be accounted for when using the matched grating interrogation method by carefully selecting gratings that are well paired [155].

The optical fibre sensors were cleaved at the tip at 90º, using the same cleaving process as the plain optical fibres, see above. As the FBGs were written in the stripped section of optical fibre, the finished product had a length of approximately 10 mm at the tip without the protective coating, consistent with the treatment of the plain fibres. In principle, the stripped section can be re-coated if required, but this was not deemed necessary for the purpose of these mechanical tests.

Once the FBG sensor has been cleaved, it may be subject to the Fabry-Perot effect. The Fabry-Perot effect generates noise on the reflected spectrum caused by interference between multiple reflections of light at the boundaries, in this case from the FBG and the boundary between the end of the fibre and the air [156]. This effect is shown in Figure 2.8. The blue line shows the spectrum before cleaving. The black line (with dots) shows the spectrum after cleaving. As can be seen in Figure 2.8 the “after cleave” spectrum is noisier than the “before cleave” spectrum, with a series of periodic fringes superimposed on the spectrum.

The noise on the spectrum could impact the sensitivity and reliability of the system by making it difficult to accurately determine the shift in the reflected spectrum. The matched FBG system relies on a predictable and stable reflected intensity from the FBG sensor. For this reason it is important to try to remove this noise as much as possible. In order to achieve this, the end of the optical fibre was painted using a black Pentel paint marker, to try and reduce the amount of reflection. This solution was chosen as it doesn’t effect the overall shape or properties of the optical fibre. The results of this are shown by the red dashed line in Figure 2.8. The results show good reduction in the noise on the spectrum. As a result, all FBG sensors used in this thesis were cleaved and painted at the tip.

2.4 CALIBRATION OF FBG SENSORS

The FBG sensor is designed to detect force at the tip of the fibre sensor. In order to achieve this, the system must be calibrated. The calibration allows any wavelength shift in the reflected light from the sensor to be related to a change in the measurand. The FBG sensor requires both a temperature and force calibration. Even though the sensor is designed to
Figure 2.8: The spectrum of the FBG sensor before cleaving (blue) and after cleaving (black). After cleaving noise due to the Fabry-Perot effect can be seen as a periodic fluctuation in the spectrum. The red line depicts the spectrum of the FBG sensor after painting the end of the optical fibre. This reduces the presence of periodic fluctuations.
detect force, it is still sensitive to temperature changes, according to Equation 1.6. Therefore the sensor must be temperature calibrated. The temperature calibration is used to correct the force measurements for any temperature fluctuations, and also to determine the temperature setting at which the matched grating interrogation system is most sensitive. The FBG sensor system has a heater element which can be used to change the temperature of the reference FBG.

2.4.1 Temperature Calibration

Temperature calibration was performed by recording and plotting the output of the FBG sensor system at different reference FBG temperatures. The ratio of the voltage output, from the sensor FBG and the reference FBG is plotted against the temperature. An example of a temperature calibration for one sensor (Sensor 1) can be seen in Figure 2.9. The ratio is recorded to help eliminate fluctuations in light intensity in the system due to changes in ambient temperature. The temperature of the room is also recorded and used to correct the data for changes in room temperature during the calibration. All testing was completed in a temperature controlled lab, and as a result only small changes in room temperature were observed. The gradient of the corrected data is recorded and used to temperature correct the force data. At different temperature differences, between the matched gratings, the baseline ratio between the sensor and reference FBGs will be different (due to the amount of matching), affecting the sensitivity of the force measurement. It is therefore important to make sure that the force calibration is completed for each sensor used with the reference temperature set to a temperature where the temperature calibration is linear. It is also important to make sure that the chosen temperature is well in the linear range so that the two FBGs are still well matched when a force is applied to the sensor FBG. This means that for optimisation of the sensor system, the temperature calibration is used so that the force calibration is performed at a temperature where there is a good linear relationship between force and output, with a steep gradient (highest sensitivity). Based on the results of the temperature calibration, force calibration and mechanical testing were completed with the reference FBG held at a temperature of 15 °C.
Figure 2.9: Temperature calibration of a 125 µm diameter FBG sensor (Sensor 1) used during testing of the dynamic mechanical properties of PDMS.
2.4.2 Force Calibration

Force calibration of the FBG sensor was performed using a custom made micrometre controlled translation stage setup. The stage was used to move the sensor to make contact with a Mark-10 5i (force resolution 0.1 mN) pressure plate force sensor at different forces. Force calibration was completed with the reference FBG at 15°C as mentioned above. The room temperature was 23°C. The force and intensity ratio (FBG sensor output) was recorded and plotted. Ratios were temperature corrected using the temperature calibration. The results of the force sensor calibration for the same sensor as the temperature calibration (Sensor 1) can be seen in Figure 2.10. The force calibration allows the output of the FBG sensor to be related to the reaction force arising from the viscoelastic material being tested. The FBG sensor system has about a 1 mN force resolution [130].

![Force calibration graph](image)

Figure 2.10: Force calibration of a 125 µm diameter FBG sensor (Sensor 1) used during testing of the dynamic mechanical properties of PDMS.
2.5 MECHANICAL TESTING OF OPTICAL FIBRES

Two types of mechanical testing of the optical fibres were performed: deflection (transverse) force testing and buckling (longitudinal) force testing in order to understand the forces that could be applied to the optical fibre before it bent or buckled. All mechanical tests were performed using three different diameters of optical fibre, these were 50 µm diameter (Fibercore, SM1500), 80 µm diameter (Newport Corporation, F-SBD) and 125 µm diameter (Corning, SMF-28). For most of the testing procedure (except when testing the effect of the coating) the protective acrylate coating was left on the optical fibres (i.e. not stripped), except at the tip of the fibre, see Section 2.3. This replicates the optical fibre containing an FBG.

2.6 DEFLECTION TESTING OF OPTICAL FIBRES

2.6.1 Theory

The deflection force \( F_D \) is the transverse force applied to the optical fibre that causes the fibre to deflect a certain distance, \( \delta_{\text{max}} \), as shown in Figure 2.11. The cantilever beam theory [157] for a concentrated load, \( F_D \), applied on the beam at any point, \( l \) from the fixed point, can be used to model the deflection force of an optical fibre.

The cantilever beam theory states that the deflection force, \( F_D \), for a given deflection, \( \delta_{\text{max}} \), applied at a location \( l \) along a beam of length \( L \), can be calculated using [157]:

\[
F_D = \frac{6EI\delta_{\text{max}}}{l^2(2l + 3b)}, \tag{2.5}
\]

where \( E \) is the Young’s modulus (72.5 GPa for silica fibre [150]) and \( I \), the moment of inertia, is given by

\[
I = \frac{\pi d^4}{64} \tag{2.6}
\]

for a cylindrical beam of diameter \( d \). Figure 2.11 depicts these parameters to aid understanding, including the length parameter \( b \). Figure 2.12 shows images of the experimental setup used for the deflection force testing. The stiffness of the optical fibre is directly related to the deflection force, as the stiffness of the optical fibre contributes to its tendency to resist the force of deflection.
The cantilever beam theory is based on a small bending approximation which is not completely consistent with this work. In particular, the theory assumes a very small bending angle, whereas the angle used here (up to 30º) is relatively large. The optical fibre doesn’t have uniform beam stiffness and diameter as assumed in the theory and the theory neglects the effects of friction (between the optical fibre sensor and force sensor probe). The diameter of the optical fibre is reduced at the tip to 125 µm once the fibre is stripped instead of 245 µm at the fixed non-stripped end. A more general theory was derived by Peter J Cadusch [131] to try to better match the actual test arrangement. This theory is referred to as the derived theory for the rest of the thesis. More information on the derived theory can be found in the supplementary material in reference [131].

2.6.2 Experimental Method

The theory of a cantilever beam with a load concentrated at varying points along the beam (i.e. an optical fibre) [157] was used to design the deflection force test set up. The optical fibre sensor was held horizontally at the base using a custom fixture as shown in Figures 2.11 and 2.12. A vertical force was applied to the optical fibre at varying dis-
2.6 Deflection Testing of Optical Fibres

tances, \( l \) from the fixed point with a pressure plate force sensor (Mark-10, 5i). The pressure plate force sensor had a force resolution of 0.1 mN. A constant overall deflection distance, \( \delta_{\text{max}} = \frac{L}{2} \) was chosen so that the deflection angle approached 30° as \( l \) approached 0. The free end of the optical fibre was deflected to this position for each load point (\( l \)). This is the same deflection angle used by Rebscher et al. in their paper on the stiffness properties of cochlear electrode arrays [154]. The magnitude of the force required to deflect the optical fibre to the constant deflection position at different values of \( l \) was recorded. This process was repeated at least four times at each load point (\( l \)).

![Image of deflection force testing on optical fibre](image.png)

Figure 2.12: Two images taken during deflection force testing on optical fibre. In these tests a wedge force sensor attachment was used to deflect the optical fibre by applying a load at different points (\( l \)). The images show testing at two different sample points.
2.6.3 Results

The results from the optical fibre deflection force tests are shown in Figure 2.13, with each point representing the mean of at least four recordings. The error bars represent the standard deviation of the recorded data. It is notable that the error bars increase for the 50 µm data at lengths greater than 8 mm. This is due to the smaller forces recorded here which are at the limit of sensitivity of the force sensor used. For comparison purposes both the cantilever beam theory, Equation 2.5 (solid line), and the derived theory (dashed line) are shown as curves on the same graph. The results show a relatively poor agreement with the cantilever beam theory, but the derived theory has an improved fit especially at the higher load locations. The experimental results are in agreement with the general trend of the theoretical curves. The magnitude of the results do not have as good an agreement with the predicted values. The derived theory appears to be closer to the recorded results for all recorded data. The diameter of the optical fibre had a significant effect on the stiffness properties of the fibres tested. The stiffness of the optical fibre decreased with reduction in the diameter of the optical fibre, for all load locations. The force required to deflect the fibre also decreased as the load concentration location increased. This means that the longer the exposed part of the optical fibre sensor, the more sensitive it is to transverse forces.

Figure 2.14 compares the deflection force measurements for a stripped optical fibre (125 µm) and plain (acrylate coated) 125 µm diameter fibre (245 µm diameter with acrylate coating). Little difference is seen between these results. The theory curves shown in Figure 2.14 predict a small difference in the magnitude of the deflection force between the stripped and non-stripped fibre, with the plain fibre predicted to be stiffer than the stripped fibre. The theory suggests that these differences are relatively small and may be lost in the error of the recorded values or be obscured by differences in friction between the glass and polymer coating. Similar to the previous figure, Figure 2.14 shows good agreement with the overall trend of the theory curves, with the theory predicting a slightly higher deflection force than the data for small load locations. However at larger load locations there is a reasonably good quantitative agreement.
Figure 2.13: The results of deflection force testing for three different diameters of optical fibres (125 µm, 80 µm and 50 µm), together with theoretical curves (cantilever beam theory = solid line, derived theory = dashed line). The error bars represent the standard deviation of multiple tests.
Figure 2.14: Results of 30° deflection force tests for a 125 µm optical fibre with coating and stripped 125 µm optical fibre. The results are compared to the derived theory for each (dashed/dotted lines respectively). The error bars represent the standard deviation of multiple tests.
2.7 BUCKLING OF OPTICAL FIBRES

2.7.1 Theory

The buckling force is the compressive force in the longitudinal direction required to buckle/bend a beam. In the case of this thesis the optical fibre will act as the beam (see Figure 2.15).

![Figure 2.15: The fixed-pinned test configuration used for buckling force tests. This condition best represents the optical fibre force sensor in use. Image derived from [131].](image)

The bifurcation buckling force theory [158] can be used to model the buckling force ($F_B$), according to:

$$F_B = \frac{EI\pi^2}{l_e^2},$$

where $E$ is again the Young’s modulus and

$$l_e = kL,$$
is the effective length, which depends on the end support conditions of the optical fibre.

Common end conditions are shown in Table 2.1. The end conditions have an effect on the shape of the optical fibre when it buckles. Table 2.2 shows how the different combinations of end conditions shown in Table 2.1 affect the shape of the optical fibre as it buckles. Of the conditions shown in Table 2.2, the condition that best represents the optical fibre force sensor when in use is the fixed-pinned condition, i.e. rotation and translation-fixed at one end, but rotation-free and translation-fixed at the other. A more detailed graphic of this condition is shown in Figure 2.15. For this situation \( k = 0.7 \). The moment of inertia \( (I) \), is given by Equation 2.6.

<table>
<thead>
<tr>
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<th>Image</th>
<th>Condition</th>
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<td>Rotation and translation fixed</td>
</tr>
<tr>
<td>Pinned</td>
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<td>Rotation free and translation fixed</td>
</tr>
<tr>
<td>Free</td>
<td><img src="image" alt="Free" /></td>
<td>Rotation and translation free</td>
</tr>
</tbody>
</table>

Table 2.1: Examples of common end conditions of the optical fibre (beam). These conditions effect how the optical fibre will buckle [158].

2.7.2 Experimental Method

The bifurcation buckling theory [158] was used to design the buckling force testing jig and procedure. The approach used was similar to the basic experimental procedure implemented by Patrick and MacFarlane [153]. An optical fibre chuck was used to secure the fibre vertically. This provided the “fixed” point. A Labview controlled Newport ILS
<table>
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<tr>
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<th>Image</th>
<th>$k$ Value</th>
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<tbody>
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</tr>
<tr>
<td>Fixed-Pinned</td>
<td><img src="image" alt="Fixed-Pinned" /></td>
<td>0.7</td>
</tr>
<tr>
<td>Pinned-Pinned</td>
<td><img src="image" alt="Pinned-Pinned" /></td>
<td>1.0</td>
</tr>
<tr>
<td>Fixed-Free</td>
<td><img src="image" alt="Fixed-Free" /></td>
<td>2.0</td>
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Table 2.2: The effect of different end conditions on the bending of an optical fibre. The dashed line represents the buckled condition [158]. The $k$ value is used to calculate the effective length of the beam, according to Equation 2.8.
100 PP translation stage, powered by a Newport EPS 300 driver, lowered a pressure plate force sensor (Mark-10 5i, 0.1 mN accuracy) with a flat compression plate attachment onto the optical fibre. This plate created a “pinned” point. For more information on the programming required to lower the pressure plate force sensor onto the optical fibre, please refer to Supplementary Material Section A. The pressure plate force sensor was lowered onto the optical fibre until it buckled. A USB oscilloscope (Stingray, DS1M12) was used to gather data during the buckling tests from the pressure plate force sensor at a sampling interval of 12 ms (the maximum rate of the force sensor output). The rate at which the pressure plate force sensor was moved towards the tip of the optical fibre was 0.2 mm/s. This speed was chosen to allow the testing to be completed in a timely manner while ensuring adequate sampling resolution during buckling, when a rapid force change occurs. Data was collected using the 3 different diameters of optical fibre (125 µm, 80 µm and 50 µm) at varying lengths (length of optical fibre exposed from the top of the fibre chuck).

Cleaving the tip of the optical fibre as described in Section 2.3 is important to achieve repeatable results. Poor contact between the flat compression plate applying the load and the optical fibre due to rough or uneven surfaces was found to create variability in the results (see Figure 2.16). There was also a concern that without an accurate 90º cleave at the tip of the optical fibre, the compressive load may not be distributed uniformly across the tip of the fibre. The 90º cleave is also consistent with the FBG sensor configuration.

Initially after cleaving the optical fibre there was still a large amount of variability in the results. Not only was there variability in the recorded buckling force, but there was also variability in the shape of the force-time curves (raw data). Figure 2.16 shows some typical variability in the results. All four curves in Figure 2.16 represent different data sets recorded on the same day with all variables kept constant. It was confirmed that the optical fibre tip was smooth. The surface roughness of the compression plate was imaged using a 3D profilometer and confirmed as the source of the variability. Figure 2.17 shows the surface roughness of the compression plate. The images show a large amount of roughness (maximum amplitude of about 40 µm), probably due to the machining process. This roughness cannot be seen with the naked eye and would probably have little effect on the results for most applications, but due to the period of the roughness being at a similar scale to the optical fibre, it was contributing to variability in the results. It is postulated that the fibre was either pivoting on the peaks or slipping down into the valleys, thereby creating variability in results.
Figure 2.16: A selection of some of the buckling force curves recorded before polishing the compression plate. These curves show the variability in the initial recorded data. Each coloured line represents a new data set. The time of initial contact is arbitrary and depends on the manual setting of the initial position.
Figure 2.17: 3D profilometer images of the compression plate roughness before it was polished, showing (a) the central region and (b) a full transverse section. The scale bar on the right hand side of the images represents the vertical height in micrometers.
The compression plate was polished to a mirror finish (final polish with a colloidal silica suspension of 0.05\(\mu\)m), which significantly improved the repeatability of the recorded results. After polishing the compression plate, the shape of the curves and buckling force were much more consistent. These results are shown in Figure 2.18. The surface roughness of the force plate after polishing is shown in Figure 2.19. This image shows a decrease in surface roughness to approximately 0.3\(\mu\)m, which resulted in good repeatability in the recorded buckling data.

Figure 2.18: Buckling force curves for data obtained after polishing. The data shows good repeatability both in the shape of the curve and the size of the buckling force. The different coloured lines represent different repeated tests. Data recorded using 80\(\mu\)m optical fibre with an exposed length of 11.2 mm.
Figure 2.19: 3D profilometer image of the surface roughness of the compression plate after polishing, showing a complete transverse section. The scale bar on the right hand side of the images represents the vertical height in micrometers.

2.7.3 Determining the Buckling Force from Experimental Data

The buckling force was considered to be the point where the critical buckling stress was exceeded, i.e. the buckling force was defined at the point where the first sign of buckling was observed [153]. This is seen as the maximum turning point of the recorded force-time graph. The optical fibre was held vertically as described in Section 2.7.2. The pressure plate force sensor was lowered onto the optical fibre at a constant speed of 0.2 mm/s. The stage was programmed to move a total of 2 mm starting from a point just above the fibre and before making contact with it. This distance was sufficient to ensure that buckling occurred. The starting point above the fibre was approximately the same for all fibre lengths. The force data collected during the testing was plotted against time as shown in Figure 2.20. The buckling force, represented by the cross, was determined to be the maximum force at the turning point of the graph. The Matlab peak finder m file was used to determine this force.
2.7 Buckling of Optical Fibres

Figure 2.20: Raw buckling force data, depicting how the buckling force was determined. The buckling force is represented by the blue cross and the dashed line. Data recorded using 125 μm optical fibre with an exposed length of 11.9 mm.

2.7.4 Results

The results of the buckling force tests are shown in Figure 2.21, for fibre lengths between 9 mm and 20 mm (125 μm fibre) and 7 mm and 18 mm (80 μm and 50 μm fibre). As the fibre diameter decreased, the force required to buckle the fibre also decreased, whereas shorter fibres can support a larger force before buckling. The curves shown in Figure 2.21 represent theoretical curves from the bifurcation buckling theory (Equation 2.7). The recorded data and the theory have good agreement for all three diameters of optical fibre (125 μm, 80 μm and 50 μm), with $r^2$ values of 0.799, 0.928 and 0.717 respectively. The $r^2$ values were determined using Matlab curve fitting tool (cftool) and Equation 2.7. All parameters were fixed.
2.8 Buckling force recorded using an FBG sensor

2.8.1 Theory

Buckling tests were also performed using SMF-28 optical fibre containing an FBG (optical fibre sensor) instead of the plain optical fibre. This was done to better understand the effect that buckling has on the output of the optical fibre sensor. The optical fibre sensor is designed to determine the force at the tip of the sensor. When the optical fibre containing the FBG has buckled, the force on the fibre is no longer aligned with the longitudinal axis of the FBG sensor. As a result it is hypothesised that the measured force will be reduced according to the force component along the longitudinal axis of the fibre. It is also predicted that the reduction in force will be related to the cosine of the angle of the buckled fibre. This relationship is shown by:

\[ F_{FBG} = F_0 \cos(\theta_B) \]  

(2.9)
where \( F_{\text{FBG}} \) is the force measured by the FBG sensor, \( F_0 \) is the applied force and \( \theta_B \) is the buckling angle defined in Figure 2.15.

### 2.8.2 Experimental Method

The method for this test was the same as the method used in the buckling tests (see Section 2.7.2) except that the testing was performed on an optical fibre containing an FBG instead of plain optical fibre. The characteristics of the FBG used in the buckling testing were: centre wavelength of 1560.26 nm, a FWHM of .51 nm and a reflectivity of -10.3 dB. This is the same sensor (Sensor 1) that was calibrated in Section 2.4. For more information about FBGs refer to Sections 1.6 and 2.3.1. In order to obtain results the FBG sensor was buckled to approximately 35° (as measured after testing using image analysis). This process was repeated 15 times.

### 2.8.3 Results

The average results of the FBG sensor buckling tests are shown in Figure 2.22. The blue dashed line shows the results obtained using the pressure plate force sensor using the same method seen in Section 2.7.2. The solid black line is the result from the FBG sensor. It can be seen that the black line (solid) is lower than the blue line (dashed) after the buckling point, as expected. The average force, seen by the force sensor after buckling (blue line) is 42.2 mN. The average force as seen by the FBG sensor (black line) for the same section is 34.4 mN. Using Equation 2.9 the expected value for an angle of 35° is 34.6 mN. This is consistent with the results obtained. Note there is some drift over time and noise in the FBG sensor data; this is expected due to the measurement system used and represents the error associated with less than 0.5 °C temperature change. Please see Equation 2.4 for more information of the relationship between temperature and force. Using the FBG sensor in the human body instead of air would potentially reduce the temperature fluctuations and help to reduce the drift seen in the output. It is important to note that for short periods i.e. where there is a great increase in force over a short period of time, the FBG sensor is very reactive and follows the expected trend. The buckling force recorded with the FBG sensor was 41.8 mN and 41.7 mN for the pressure plate force sensor. These values are very similar; this was predicted as this is the point where the fibre started to buckle. The pressure plate force sensor and FBG sensor should follow the
same trend until this point. The force measured by the FBG sensor is only affected by buckling after this point.

![Graph showing force over time for FBG and Force sensors](image)

**Figure 2.22**: Results of the buckling data (average of 15 repeats) gathered with the FBG sensor. The graph shows both results from the pressure plate force sensor (blue dashed line) and the optical fibre sensor (black line). Data recorded using 125 µm optical fibre with an exposed length of approximately 20 mm.

## 2.9 Conclusions

Manufacture and calibration of the FBG sensor was performed as described in this chapter. This allows the FBG sensor to be used as a force sensor, to measure dynamic mechanical properties. Calibration also helps to maximise performance of the sensor.

The results of the deflection and buckling force tests provide a better understanding of the maximum transverse and longitudinal forces that can be applied to the optical fibre sensor before its performance becomes compromised. The optical fibre sensor, which has been designed to determine the dynamic mechanical properties of viscoelastic solids, contains 10 mm of exposed 125 µm optical fibre. Using the results from the deflection (Figure 2.13) and buckling tests (Figure 2.21) it can be concluded that the maximum forces
that should be applied to that sensor are 7 mN (transverse) and 210 mN (longitudinal). These values are length dependent. If the length of the exposed fibre was decreased, the force required to buckle and bend the fibre would increase.

The deflection forces of the optical fibres followed the expected trend, with greater force required to bend the fibre as the diameter increased. This is consistent with the theory and results for all three diameters of optical fibre tested (Equation 2.5). Larger forces could be applied to the optical fibre at smaller distances from the fixed point i.e. as the load location moved towards the fixed point, the deflection force increased. However the results did not show exact agreement with the magnitude of this theory. The recorded results were mostly lower than those predicted by the theory. It is thought that a combination of assumptions including uniform diameter and absence of friction could be the reason for this. Removing the protective coating from the 125 μm diameter optical fibre had little effect on the recorded results. The theory predicted a small decrease in the magnitude of the results when the coating is removed. Once again, this shift may be obscured by changes in friction between the wedge pressure plate force sensor attachment and the two different fibre materials.

The results of buckling force tests on optical fibres followed a similar trend to the deflection force tests in which the force required to bend the optical fibre decreased as the diameter of the embedded optical fibre decreased. Also the buckling force decreased as the length of the optical fibre increased. The experimental data was found to agree well with the results predicted from the theory (both in trend and magnitude).

With the incorporation of the FBG sensor, it was possible to demonstrate how the output of an FBG force sensor can be affected by buckling. Although the buckling force recorded by the FBG sensor followed the predicted theory, the results after the buckling point are less predictable and would rely on a detailed knowledge of the bending angle and friction between the fibre and force plate (not always possible). The results support the conclusion that buckling of the FBG force sensor should be avoided during measurements.

When considering the use of optical fibre sensors for any application, the transverse and longitudinal compressive forces on the sensors need to be considered. The data provided in this work provides indications of the maximum force that can be exerted on an optical fibre sensor for a given length and fibre diameter before potentially detrimental bending/buckling occurs.
DETERMINING DYNAMIC PROPERTIES OF MATERIALS USING DYNAMIC MECHANICAL ANALYSIS (DMA)

3.1 INTRODUCTION

Although there is limited reliable data in the literature about the dynamic mechanical properties of tissue and how they change when they become diseased, it is widely understood that there are changes (see Section 1.2). Much of the data available refers to static measurements. Many references simply state that the mechanical properties of tissue change with tissue health, without reference. Despite an extensive literature review, it appears that the frequency response (stiffness) of a number of materials including biological materials is not well characterised. It is conceivable that some materials have been measured, but that the results are held as industry proprietary information.

As part of the testing of the FBG sensor there would ideally be some standards to compare it against to make sure that the results are valid. Therefore the aim of this chapter was to evaluate the DMA technique so that results obtained using DMA could be compared with those achieved using the FBG sensor to ensure that the obtained results are consistent. For more information on DMA please refer to Section 1.3. DMA data was obtained for both the PDMS samples which approximate the viscoelastic properties of human tissue, and for optical fibre. PDMS was chosen as a DMA test material, as it is a viscoelastic solid that mimics some of the properties of human tissue. PDMS can also be easily formulated with different stiffnesses. It was found that currently there is an interest in measuring the dynamic mechanical properties of human tissue using DMA [37, 34, 35, 16, 38]. The data reported in these articles for various human tissue samples may prove valuable in future work if the FBG sensor is applied in vivo (not part of this thesis). In that case the FBG sensor could be tested using these materials and compared to the published results. Unfortunately no DMA results were found for PDMS of varing
stiffness. However, static results are available [15, 22] as well as dynamic results using a slightly different method [17]. Some data is available for comparison with the DMA results for optical fibre [32].

3.2 Dynamic mechanical properties of PDMS

PDMS is a type of silicone elastomer that has been used to mimic tissue properties [22]. Some papers have shown that by altering the base:crosslinker ratio used to make PDMS, the Young’s modulus can be modified [15, 22]. This is shown in Figure 3.1.

![Bar graph](image)

Figure 3.1: Bar graph representing how the crosslinker ratio affects the Young’s modulus of PDMS. Image reproduced from [15].

Using a custom-made Teflon mould, PDMS disks with four different crosslinker ratios were produced. The PDMS disks were 15 mm in diameter and 5 mm thick. The four different crosslinker ratios used were 10:1, 20:1, 40:1 and 50:1. The 10:1 ratio is a common ratio used with PDMS. The 40:1 and 50:1 ratio were used to create samples with a similar Young’s modulus to human muscle tissue based on information found in the literature [15, 22]. The 20:1 ratio PDMS lies in the middle of the PDMS modulus range.
It should be noted that due to the complex nature of human tissue, no tissue phantom can be expected to demonstrate exactly the same viscoelastic behaviour as biological tissues. This is mainly due to the inhomogeneity and anisotropy of tissue on the range of length scales. As discussed in Section 1.4, the measurement method and frequency at which the Young’s modulus of viscoelastic materials is determined can show a large amount of variability [15]. Also there is a natural variation in the stiffness of human tissue between people and with age. These issues mean that great care is needed to even define what is meant by “similarity” in the context of tissue phantoms. In practise, the validity of the tissue phantom may come down to the subjective tactile impression of a skilled practitioner. Given these limitations, these homogeneous PDMS samples were used for convenience in this work to evaluate the DMA technique and for initial device testing.

When making the PDMS disks the base and crosslinker used to make up the PDMS was first made up in the correct ratio (care was taken to make sure that the two components were properly mixed) and the whole mixture was then degassed for at least 30 minutes. After degassing the mixture was poured into the moulds. In order to get a smooth finish the moulds were over filled and excess mixture was then removed by scraping a glass microscope slide over the top of the mould. The mixture was then degassed again while in the mould. After the second degassing the mould was then placed in the oven preheated to 80°C, to cure overnight. It is important to make sure the PDMS is correctly degassed as bubbles in the PDMS disks could lead to artefacts in the DMA results. It is also important to make sure that the PDMS is mixed thoroughly; otherwise it can lead to inhomogeneous samples. An image of one of the PDMS disks used in the DMA testing is shown in Figure 3.2.

3.2.1 Experimental Method

The PDMS disks (10:1 and 50:1 ratio only) were tested in the TA Instruments DMA 2980 using the small disk compression clamps as shown in Figure 3.3. The PDMS disks were the same diameter as the clamp disks (15 mm). All PDMS testing was completed using a frequency sweep from 150 Hz down to 1 Hz at 35°C (testing was performed inside a temperature-controlled furnace) with an amplitude of 15µm and preload of 1 N. These parameters were a compromise between the parameters planned for the optical fibre
sensor and those required for stable operation of the DMA testing system. The frequency range was based on the operating limits of the DMA.

The 10:1 ratio PDMS was tested over three successive days to check the consistency of the results. The 50:1 data was only examined on the first day of testing. The 20:1 and 40:1 ratio PDMS were not tested with the DMA due to time constraints and issues encountered in obtaining reproducible results with the other ratios.

### 3.2.2 Results

The DMA results for PDMS are shown in Figures 3.4 to 3.6. The modulus shown was calculated using the DMA software based on the DMA theory defined in Section 1.3. The results in Figure 3.4 (storage) and Figure 3.5 (loss) show the average data taken from a number of samples. For the 10:1 ratio PDMS, six samples were tested, with repeats performed on three testing days. The data in Figure 3.4 and Figure 3.5 shows the average of all 18 data sets. The 50:1 ratio PDMS was tested using nine samples. All of these samples were tested on the same day. The results shown in Figure 3.4 and Figure 3.5 represent the average of all nine data sets. The shaded regions shown in Figure 3.4 and Figure 3.5 represent the spread of the data (mean ± standard deviation).

The variability in the results recorded for the 10:1 ratio PDMS over three days is shown in Figure 3.6 (storage) and Figure 3.7 (loss). Each of the storage or loss plots represents a
Figure 3.3: Image of the DMA compression clamps used to test PDMS samples.
Figure 3.4: DMA results showing the storage modulus for 10:1 and 50:1 ratio PDMS. The shaded regions represent the spread of data (mean ± standard deviation).

Figure 3.5: DMA results showing the loss modulus for 10:1 and 50:1 ratio PDMS. The shaded regions represent the spread of data (mean ± standard deviation).
different day for data recording, as labelled, and each sample is represented consistently by the same colour trend line in each graph. The Day 1 plot of the storage modulus corresponds to the same day of testing and the same samples as the Day 1 plot of the loss modulus, and similarly for the Day 2 and 3 plots. There is no apparent pattern in the results, with the different samples giving highly variable results within a broad range, depending on the day of measurement. Please note that a great deal of care was taken to ensure that the individual sample measurements where completed in a repeatable manner.

3.2.3 Discussion

The results obtained show significant variability in the moduli (Figure 3.4 and Figure 3.5) and differences in results recorded on different days (Figure 3.6 and Figure 3.7). For the 10:1 ratio PDMS samples there is an average standard deviation in the storage modulus of 0.55 MPa across the full data set (Figure 3.4) and an average of 0.53 MPa on the individual days (Figure 3.6). Comparison of these results suggests that the inconsistent results are mostly due to the measurement technique and testing equipment rather than environmental factors or day-to-day variability in the PDMS sample. Therefore it is difficult to draw any conclusions about the material tested. The results shown in Figure 3.4 and Figure 3.5 show an overlap in the error bars of the data. There is a notable difference in the stiffnesses (by feel) of the two sample types due to the different crosslinker ratios. The appropriate testing device should be able to measure this difference more reliably. It can be seen that there is an increase in the spread of the storage modulus for the 10:1 ratio PDMS compared to the 50:1 samples. This increase in data spread may be due to the increased number of measurements, including results from multiple days. The size of the error might also scale with the size of the storage modulus, although the relative error is also larger for the 10:1 samples than for the 50:1. Day to day DMA variability is shown in Figure 3.6 (storage) and Figure 3.7 (loss). The data in Figure 3.6 and Figure 3.7 show significant differences in the results when the only changing variable was the day of data collection.

When compared to the previous published data shown in Figure 3.1, the average result of the 10:1 ratio PDMS was lower (1.0 MPa compared to 1.1 MPa) but within the error, whereas the average result for the 50:1 ratio PDMS was higher (0.4 MPa compared to 0.07 MPa). However, some caution must be exercised when comparing the present results
Figure 3.6: Repeated DMA recordings showing the storage modulus for a set of 10:1 ratio PDMS samples over 3 different days. Each colour represents the result from the same PDMS sample. Note that different samples gave high or low measurements, depending on the day.
Figure 3.7: Repeated DMA recordings showing the loss modulus for a set of 10:1 ratio PDMS samples over 3 different days. Each colour represents the result from the same PDMS sample. These are the same samples and colours as presented in Figure 3.6. Note that different samples gave high or low measurements, depending on the day.
with the static moduli reported in reference [15]. The dynamic modulus consistently appears to decrease as the frequency approaches zero, but it would be unrealistic to try and extrapolate to zero, given the overall uncertainty of the results. It was also noted that the DMA machine used here is specified for materials with elastic moduli between 0.5 MPa and 100 GPa [159]. The data for 10:1 ratio PDMS is just within this range while the 50:1 ratio PDMS is below this range. Given that the elastic moduli of the PDMS are at the bottom end of the specified range for the DMA, this could contribute to the variation seen in the results.

3.3 Dynamic Mechanical Properties of Optical Fibre

The viscoelastic properties of the optical fibre were tested to understand the contribution of the optical fibre sensor to the overall output of the FBG sensor system and to demonstrate that the optical fibre is much stiffer than the tissue to be tested. For the FBG sensor to work effectively, the optical fibre containing the FBG will need to be pushed against the tissue. The system measures the sum of all the displacements. If the optical fibre is much stiffer than the material tested, it will have minimal displacement, and will therefore have minimal effect on the results. In order for the system to work as required, the tissue (or material being tested) must be the least stiff material. The stiffness of the measurement system has been referred to as the “load frame compliance” [87].

3.3.1 Experimental Method

The dynamic properties of optical fibre were tested with the same TA Instruments DMA 2980 as Section 3.2, but using the tension clamps as shown in Figure 3.8. Optical fibre samples (125 μm - Corning, SMF-28) were glued between two brass plates (10 mm x 10 mm x 1 mm) at each end using ProSciTech crystalbond 509. The configuration of the optical fibre between the metal plates can be seen in Figure 3.9.

This method is based on the approach used by Stefani et al. [32], where the dynamic properties of a number of optical fibres were investigated. The DMA fibre clamps were originally used, however it was found that they could not hold the optical fibre securely due to its small diameter. The metal plates allowed the DMA tension clamps to hold the optical fibre securely, despite the relatively small diameter. The 250 μm polymer coating of the optical fibre was mechanically stripped (see Section 2.3) and the fibre was tested
The optical fibre was tested at two lengths, 25 mm (14 samples) and 15 mm (15 samples), measured between the inner edges of the metal plates. The fibre was tested using an amplitude of 15 µm and a preload of 1 N (pressure of 5.7 kPa). The furnace was not used during optical fibre testing. As a result all data was taken at room temperature.
Figure 3.9: Image of an optical fibre sample used for DMA testing. The optical fibre is glued between metal plates to allow stable positioning in the DMA.
3.3.2 Results

The DMA results for the 25 mm optical fibre can be seen in Figure 3.10. Similar to the results shown for the PDMS samples, the displayed modulus was calculated using the DMA software. The results show what appears to be system resonance starting around 70 Hz. It can also be seen in Figure 3.10 that the values obtained at frequencies of 50 Hz or less are relatively constant. The mean storage modulus (real part) for this data (less than 50 Hz) is 66.2 GPa with a standard deviation of 0.5 GPa. It was postulated that the resonance of the system was dependent on the length of the optical fibre under test. Therefore to confirm that the anomalous values were due to a system resonance, the optical fibre was also tested at a length of 15 mm.

Figure 3.10: DMA results for optical fibre samples of length 25 mm. The top line (red) represents the storage modulus and the bottom line (blue) represents the loss modulus. Error bars show the standard deviation of 14 samples.

The results of the loss and storage modulus for the 15 mm optical fibre are shown in Figure 3.11. It appears from the results that the resonance shifted to frequencies above 150 Hz. Unlike Figure 3.10 the data was relatively flat across the full range, but may have
started to drop after about 100 Hz (storage modulus). It was felt that this may be the start of the resonance at this new length, but more testing would be needed to confirm this.

![DMA results for optical fibre samples of length 15 mm. The top line (red) represents the storage modulus and the bottom line (blue) represents the loss modulus. Error bars show the standard deviation of 15 samples.](image)

The mean storage modulus over the measured frequency range is 58.4 GPa with a standard deviation of 0.2 GPa, which is not in good agreement with the result for the 25 mm fibres. However, given the variability seen in the PDMS results, this lack of repeatability was not unexpected.

### 3.3.3 Discussion

The average dynamic storage moduli obtained for the 25 mm optical fibre samples (66.2 ±0.5 GPa) and the 15 mm samples (58.4 ±0.2 GPa) are lower than the documented static modulus of 72.5 GPa [150] and the value of 71 GPa given by previously reported DMA measurements [32] for SMF-28 optical fibre. Given that the data exhibits no apparent frequency dependence in the absence of any structural resonance, the average dynamic moduli can be directly compared with the static case (zero frequency). However
it should be noted that these values are approaching the upper limit of the DMA used for testing as discussed in Section 3.2.3 [159]. The difference in results may also be due to the use of crystal bond to secure the fibre samples. The presence of crystal bond may allow some additional movement to occur in the optical fibre, which would be detected as an increase in strain and therefore a decrease in the storage modulus. Further testing is required to support this theory. It is also possible that the values are due to variations in the test procedure (preload and strain amplitude), as the results of mechanical testing are known to be influenced by the conditions used [159, 160]. However, this is perhaps unexpected in a predominantly elastic material such as glass as opposed to viscoelastic materials such as PDMS.

It was also noted that at 15 mm, the storage modulus of the optical fibre was reduced (58.4 GPa). The PDMS results (Figure 3.6) suggest that some of this shift in modulus may simply be associated with day-to-day variability in the DMA. As the optical fibre DMA results were obtained manually (without using the data acquisition software) the temperature controlled furnace could not be used. Temperature differences on multiple testing days could have affected the results. When looking at the results for the two different lengths, of interest is the apparent shift in the resonance of the system, supporting the hypothesis that the resonance is length dependent, similar to a guitar string. This hypothesis is based on the theory of a standing wave on a string [161]: as the length of the string decreases, so the resonance frequency increases in inverse proportion. This theory is only supported qualitatively by the results shown here and further investigation was considered beyond the scope of this thesis.

3.4 Conclusion

In the context of the thesis, it was intended to establish a benchmark for the behaviour of optical fibre and PDMS across the frequency range of interest. However, the outcome of the DMA testing was not as useful as originally hoped. Variability in the data made it difficult to obtain reliable quantitative results for the complex Young’s modulus of the materials tested and raises questions about the reliability of the instrument. The ASTM standards for DMA testing [159, 160] recognise that the consistency of results is reliant on maintaining identical experimental conditions. This could help to account for differences between the results reported here and those published in the literature. It is also noted that results can be influenced by a range of mechanical factors and physical characteris-
tics that relate to the load frame compliance of the measurement system. These factors include stiffness, damping, accuracy of loading, alignment, thermocouple location and temperature instability. Lack of transparency in the calibration procedure is a further concern. These issues highlight potential issues with the age (over 20 years) and condition of the DMA used, as it is no longer fully supported by the manufacturer. Material factors including quality, consistency and measurement errors can also influence the outcome [160, 19]. The poor repeatability of the results could be due to any number of factors including load frame compliance, temperature changes or calibration issues. As suggested by the ASTM, there is also a concern that the stiffness of the materials tested are at the limits of moduli of materials commonly tested with DMA [159].

In testing, much care was taken to remove any random and operational errors. Multiple testing configurations and parameters were considered, testing was repeated a number of times and the company technician was consulted with regards to the best testing method and calibration. Even after the best care there was still large variability in the results. It has therefore been concluded that the source of error is most likely instrumental. The instrumental error is highlighted in the results shown in Figure 3.6. These results show large random variations when the only variable is the day of testing.

The load frame compliance refers to the compliance (stiffness) of the testing system [87]. The output of the system is the sum of all the compliance in the system. To achieve the most accurate measurement of the stiffness of the material being measured, the stiffness of the testing system must be much greater than that of the material being tested, thus contributing less to the outcome. It is possible that the DMA which is most commonly used for testing polymers is not rigid enough to test stiff materials such as optical fibre. This could account for the results found in Figure 3.10 and 3.11 which are lower than the values presented in the literature.

Temperature changes could affect the measured results. When testing the PDMS, a temperature controlled furnace enclosure was used to try and reduce temperature changes; however the calibration process and the stability of this furnace are unclear. In the optical fibre testing, the furnace could not be used due to the need to manually record data. These results could therefore be affected by environmental temperature changes. It should be noted that there was about a 3°C temperature difference in the lab when the results for Figures 3.10 and 3.11 were recorded.

Good calibration is important for good results [160]. Interestingly the ASTM DMA test standard states that calibration for the storage modulus should be completed in accordance with recommendations given by the manufacturer [160]. Calibration was per-
formed in this manner, but there was a lack of transparency about the calibration process, even after consultation with the company. It is believed that the calibration of the DMA takes place at the zero displacement position. However, a recent paper in relation to nanoindentation suggests this is not the most accurate method [162]. The paper suggests that as the dynamic response is the combination of the testing system and the material being tested, it is important to understand the response of the system at the position it is being tested and at each of the frequencies to be tested. If this is the case it could contribute to the variability in the results. A better understanding of the DMA calibration procedure and the relevance of the calibration at the actual testing position and testing frequencies used is needed to confirm this hypothesis.

Although obtaining DMA data for optical fibre and PDMS proved more difficult than predicted and delivered somewhat variable results, the process has still allowed a better understanding of the materials and the difficulties faced when measuring the dynamic properties of viscoelastic materials. The difficulties faced could also contribute to the lack of literature found in this area. The main conclusion is that the optical fibre is significantly stiffer than the PDMS (~60 GPa vs ~1 MPa). This result is promising for the outcome of the bench top testing, which is discussed in the following chapter.
4

DETERMINING DYNAMIC PROPERTIES OF PDMS USING BENCHTOP OPTICAL FIBRE SENSOR SYSTEM

4.1 INTRODUCTION

Using an FBG sensor to determine the dynamic mechanical properties of viscoelastic materials is a novel approach. Therefore a benchtop setup was established to test this technique. PDMS samples with different base:crosslinker ratios were used to determine if the FBG sensor could differentiate between different stiffness properties (these were the same samples that were used in DMA testing, see Section 3.2). The benchtop system consisted of a modular piezoelectric flexure nanopositioning stage (PI P-280.20) that was used to press the FBG sensor sinusoidally onto the test sample. The sample was held between the FBG sensor and a pressure plate force sensor (used as the sample platform - see Section 4.2) as shown in Figure 4.1. A specified preload was provided to the sample to make sure that the FBG sensor did not lose contact with the sample at any point during the testing process.

4.2 BENCHTOP SETUP DESIGN

The benchtop setup takes advantage of the pressure plate force sensor (Mark-10, 5i) used in the mechanical testing of the optical fibre (see Section 2.5) and the FBG sensor described in Section 2.3.1. The pressure plate force sensor plays two roles: firstly to verify the results obtained using the FBG sensor and secondly provides a platform for the test sample to sit on. It also has the added bonus of providing useful real-time feedback during the entire testing process. The FBG sensor system was used to measure the stiffness properties of the PDMS samples tested. The FBG sensor was placed into a fibre chuck, which was mounted on a PI modular PZT flexure nanopositioning stage (PI, P-280.20).
Figure 4.1: Benchtop testing setup for the FBG sensor including schematic of system diagram (top) and real image (bottom).
The stage was driven by a Rigol DG4162 function waveform generator at different frequencies (0-20 Hz). This allows the FBG sensor to be pressed against the sample at the desired frequencies. The benchtop setup is limited to testing at frequencies lower than 20 Hz, due to the limitations of the closed loop feedback system on the PZT driver and controller (PI, E-500k012). The phase shift between the driving signal from the waveform generator and the signal recorded by the FBG sensor, together with the amplitude of the recorded signal are used to determine the stiffness properties (complex modulus) of the sample as described in Section 4.3.2. Data from the bench top system (driving signal and FBG sensor) was recorded using an AD-Instruments Powerlab 4/35.

The testing system was mounted onto an optical table using a bulky aluminium structure. Care was taken to make sure that the mounting was as rigid as possible in an effort to minimise the effect that the load frame compliance of the mounting system had on the measurements.

4.3 METHOD DEVELOPMENT FOR MEASURING DYNAMIC MECHANICAL PROPERTIES

The complex modulus of the samples tested is determined from the phase shift of the data and the peak-peak amplitude of the FBG sensor signal. As the phase shift can be determined by the distance between the peaks of the driving signal and recorded FBG sensor signal, the location of the peaks must first be determined with the highest possible accuracy. In Matlab there is a peakfinder m file (the same file that was used in Section 2.7.3) that can be downloaded and used to determine the peaks of a given data set. This file however has issues when there is noise in the data. To improve the accuracy a new code for peak detection was developed as part of this thesis. Figure 4.2 shows an example of the increased accuracy of finding peaks using the developed peak finding method. Further analysis to investigate the accuracy of this method has been completed, please refer to Section 4.3.1. The code first finds the maxima in the data using the peakfinder code. A typical maximum is represented by the circle in Figure 4.2. It then takes a section of the data on either side of each maximum point and fits a parabola to that data. The fitted curve is the black curve shown in Figure 4.2. The turning point of the fitted curve is used as the peak of the data, depicted by the cross in Figure 4.2. The peak-peak amplitude of the fitted data is also recorded as the magnitude of the FBG sensor signal. To find the peak-peak amplitude, the peak detection code was used to find both the maxima and
minima. Comparison of these two methods was completed to confirm that the developed method improved the accuracy of identifying the peaks and hence the phase shift and amplitude.

### 4.3.1 Comparing the Two Peak Finding Methods

In order to test the two peak finding methods, two synthetic sine waves were created with a signal-to-noise ratio of 15 dB. One sine wave represents the driving force, and the other, the response. The sine waves had constant known frequency of 10 Hz and phase shift of 90° (π/2). Using the peak finding methods a straight line graph of peak number versus phase should be created, with a constant phase value. Any deviation from the constant phase value is an indication of the error in the peak detection. The method with the least deviation from the constant value is the most accurate. Figure 4.3 shows
the data collected using the peakfinder method and Figure 4.4 shows the data from the developed method (the curve fitting method of peak location developed for this work).

By comparing the standard deviation for the peakfinder method ($\sigma = 10.1^\circ$), and the developed method ($\sigma = 0.9^\circ$), it can be seen that there is a significant improvement in the accuracy using the developed method. The improved accuracy of the developed method was further tested by looking at the standard deviation of the data for a number of signal-to-noise ratios in the synthetic data. These results are shown in Figure 4.5. Figure 4.5 shows a decrease in the standard deviation for each signal-to-noise ratio tested using the developed peak finding method. The error estimate shown in Figure 4.5 also appears less affected by the signal-to-noise ratio of the data. This statement is based on the reduced gradient of the data using the developed method ($0.5^\circ$/dB) compared to the peak finding method ($3.4^\circ$/dB). As a result the developed method will be used to determine the phase shift and amplitude of all data obtained using the benchtop setup.

During testing it was observed that there was some drift in the light signal of the FBG data, resulting in variations in the baseline of the data. The peak-to-peak amplitude was unaffected, as the change in peak amplitude was due to an offset, not a loss of data. This effect made it difficult for the peak detection method to determine all of the peaks, especially for the softer PDMS samples (40:1 and 50:1) where the recorded amplitude is very small. In order to overcome this issue, the data was passed through a low-pass Butterworth (fourth order) filter in Matlab before running the developed curve fitting peak detection file. This low-pass filter successfully removed the fluctuating offset allowing for the peak amplitudes to be calculated more consistently. Care was taken to make sure that the peak-peak amplitude, an important factor for calculations, was not affected. The cut-off frequency of the low-pass Butterworth filter was set to five times that of the driving frequency for each data sequence.

From Phase Shift to Complex Modulus

The phase shift between the driving signal and the FBG sensor data, together with the peak-to-peak amplitude of the FBG sensor data, is used to determine the dynamic complex modulus of the samples tested. The theory used to determine the complex modulus is based on the theory of DMA (see Section 1.3) and the theory of contact mechanics/indentation testing [86]. As shown in Equation 1.2 the complex modulus is the vector sum of the real elastic modulus ($E'$) and the imaginary viscous modulus ($E''$). The amplitude
**Determining Dynamic Properties of PDMS Using Benchtop Optical Fiber Sensor System**

Figure 4.3: Results of phase shift data using the basic Matlab peakfinder m file function. The synthetic input data had a signal-noise ratio of 15 dB.

Figure 4.4: Results of the phase shift data using a customised developed method for peak detection, for the same input data as Figure 4.3.

Figure 4.5: Comparison of the standard deviation of the data set for the basic Matlab peakfinder m file function and the developed method over a range of signal-to-noise ratios.
of the FBG sensor data is used to determine the real modulus ($E'$) and the phase shift is used to determine the imaginary modulus ($E''$). The relationship between the amplitude of the FBG sensor data and the real modulus ($E'$) is based on a flat-ended cylindrical indenter. The type of indenter used affects the relationship between the amplitude of the FBG sensor data and the real modulus ($E'$). The optical fibre used as the indenter is best represented by a flat-ended cylindrical indenter. A schematic of the flat-ended cylindrical indenter, which is representative of the FBG sensor, is shown in Figure 4.6. Please refer to

![Schematic of a flat-ended cylindrical indenter, representative of the FBG sensor.](image)

Figure 4.6: Schematic of a flat-ended cylindrical indenter, representative of the FBG sensor.

Section 1.3 for further definitions of the symbols in the following equations.

The real modulus ($E'$) is determined by [31, 86]:

$$E' = \frac{3F(1 - \eta)}{4rD_s}$$  \hspace{1cm} (4.1)

where $F$ is the peak-peak amplitude of the force measured by the FBG sensor, $\eta$ is the Poisson ratio ($\approx 0.5$), $r$ is the radius of the indenter (optical fibre) and $D_s$ is the displacement of the sample, which is determined by the oscillatory driving signal and the stiffness of the testing sample. The $D_s$ value was different depending on the stiffness of the material tested. For the 10:1 ratio PDMS the average $D_s$ value was 22$\mu$m, but increased towards 25$\mu$m with decreased stiffness.

The phase shift of the data ($\phi$), is determined by:

$$\phi = \frac{(t_j - t'_j)}{(t_{j+1} - t_j)} \times 360^\circ$$ \hspace{1cm} (4.2)

where $t_j$ represents the time value of peak $j$ (FBG sensor signal) and $t'_j$ represents the time value of peak $j$ (driving signal) as shown in Figure 4.7.
The phase shift can then be used to find the imaginary modulus ($E''$) using:

$$E'' = E' \tan(\phi)$$ (4.3)

and frequency ($f$) is given by:

$$f \approx \frac{1}{(t_{j+1}' - t_j')}.$$ (4.4)

Note that both the displacement ($D$) and the phase shift ($\phi$) must be corrected to account for the load frame compliance of the system. This is discussed in Section 4.5.

### 4.4 Linearity Testing

As this testing method uses both a PZT actuator and associated driver electronics, it is important to understand the relationship between the input voltage (driving signal/voltage) and the output (movement) of the PZT. Therefore it was necessary to calibrate the PZT. Good PZT systems will have good linearity i.e. the input voltage and the output movement will have an accurate linear relationship. Feedback systems can be used to improve the linearity of the system. The benchtop system being tested has an analogue servo controller (P-I E-509) with a 3 kHz notch filter, to help compensate for the resonance of the PZT and capacitive sensors as a feedback system. The feedback system is
designed to improve linearity and eliminate drift. The FBG sensor is pressed against a pressure plate force sensor; therefore the force is related to the movement of the PZT.

4.4.1 Experimental Method

In order to test the linearity of the system, a DC voltage supply was connected to the PZT driver. The driver was then connected to the PZT. An optical fibre chuck was attached to the PZT actuator stage and the fibre sensor was advanced up against the pressure plate force sensor with approximately 50 mN preload (Mark-10 5i). This mimics the configuration of the PZT that was used for the subsequent sample testing, i.e. the vertical orientation shown in Figure 4.1. The input voltage was then applied to the driver at 0.5 V intervals between 0 - 2.5 V. This voltage range relates to the calibrated force range of the FBG sensor. The input voltage and the force shown on the pressure plate force sensor (representative of the movement of the PZT) were recorded and plotted against each other. All linearity tests were completed with driver feedback on. This is the setting that was used in all subsequent materials testing.

4.4.2 Results

The results of the linearity test using two different FBG sensors are shown in Figure 4.8. The results show a good linear relationship between the input voltage and the resulting net force provided by the FBG sensors (measured force minus preload). The feedback system is important in achieving this linear response. It was noted that FBG Sensor 1 showed increased linearity ($r^2 = 0.999$) over the range tested compared to FBG Sensor 2 ($r^2 = 0.997$). Each FBG sensor was manufactured individually by the author, and as a result there is some variation in the FBG parameters. This results in some FBG sensors being more sensitive than others when used with the FBG interrogation system (for more information please refer to Section 2.2). It may be possible to improve the sensitivity of FBG Sensor 2 by changing the temperature of the reference FBG in the interrogation system, but this would require a recalibration of the sensor (see Section 2.4). FBG Sensor 1 was used for all reported data except when specified.
Figure 4.8: Linearity testing results for the PZT electric transducer with two different FBG sensors used in the benchtop rig. The red lines represent the linear best fit.
4.4.3 Discussion

The good linearity seen in Figure 4.8 between the input voltage and the applied force implies a linear movement of the PZT when the driver feedback is on. This is a desirable result for mechanical testing. In order to achieve this it is important that the feedback system is well calibrated for use with the PZT used. Unfortunately even though the PZT and feedback system used here worked well, they were not originally designed to work together; as a result they are not an ideal pairing. This meant that the system needed to be readjusted regularly and there was a limitation on the frequency range that could be used (roughly 0 - 20 Hz). It was noted that FBG Sensor 1 has a higher sensitivity and linearity than FBG Sensor 2. This is due to better matching with the FBG used in the interrogation system. For more information on the matched grating interrogation systems, please refer to Section 2.2. Increased FBG sensor sensitivity is preferable when testing samples; however there is always a compromise between sensitivity and measurement range.

The pressure plate force sensor provides real-time feedback about the force recorded on the FBG sensor. Real-time feedback is convenient for setting the preload of the system and to make sure that excessive force is not applied to the FBG sensor causing it to be damaged. Although it is possible to create an FBG-based sensor system where real-time feedback is directly available the current system does not provide this. Therefore it was decided that for convenience the pressure plate force sensor would remain in place for all of the tests. This raises questions about the effect of the pressure plate force sensor on the measurement system. It was understood from the work done with the DMA (see Chapter 3), that the load frame compliance of the measurement system and the measurement technique can have an effect on the results. In light of this concern, measurements were taken to determine a calibration factor. This calibration factor was taken into consideration when presenting results. A detailed explanation of the calibration process is provided in Section 4.5.

Although the effect of the pressure plate force sensor can be taken into account through the calibration factor, linearity tests were completed by recording data from the benchtop FBG sensor system when pressed up against the pressure plate force sensor (as in Figure 4.8) and against a rigid structure (with the pressure plate force sensor removed) in order to look at the effect of the pressure plate force sensor in the benchtop setup. The results of this comparison are shown in Figure 4.9. Please note that Figure 4.9 used FBG Sensor 2.
Figure 4.9: Comparison of the linear testing both with and without the pressure plate force sensor present. The red lines represent the linear best fit.

This was done to reduce the chance of FBG Sensor 1 being damaged and thus becoming unavailable for sample testing.

The results in Figure 4.9 with the pressure plate force sensor present show good linearity ($r^2 = 0.997$) as discussed in Figure 4.8. However the rigid data shows a significant change in system behaviour. The first point of note is the lack of response between 0 and 0.5 volts. This is due to the FBG sensor not coming into contact with the rigid structure until between 0.5 and 1 volts i.e. there was no preload force. Without the presence of the pressure plate force sensor, it was difficult to tell when the FBG sensor comes into contact with the rigid structure. The second interesting point is that the recorded force increases more sharply to a higher final value without the pressure plate force sensor in place. With the pressure plate force sensor in place there is only a 74.5 mN increase in the force from 0.5 - 2 volt increase. Whereas once the pressure plate force sensor is removed the force increase for the same voltage range is 236 mN. This suggests that the pressure plate force sensor is not rigid and deflects when loaded, thus reducing the overall compression seen by the FBG sensor. This result reinforces the need for a calibration step to account for the compliance of the load frame (Section 4.5). The final note is that the rigid data is not
as linear as the other results ($r^2 = 0.973$). In this regard it is important to understand that the FBG sensor system has a force range limited by the desired force sensitivity [150]. For more information on this aspect of the sensor system please refer to Section 2.2. The FBG sensor used in the project was designed and calibrated to perform with maximum sensitivity in the force range 0 - 100 mN. This was the range required for mechanical testing of the PDMS samples. The forces measured as part of the PDMS testing were on average 30 mN (see Section 4.7). The recorded forces during linearity testing at higher voltages were well outside this range, but serve to reveal the effect that the pressure plate force sensor has on the load frame compliance. What is important for the dynamic mechanical testing of viscoelastic solids is that the FBG sensor data is linear within the calibrated range.

Due to the convenience of having the pressure plate force sensor present in the measurement system, it was decided to keep it in place. In order to do this the effect of its presence must be taken into consideration during all data acquisition through calibration/correction for the load frame compliance of the complete system.

4.5 CALIBRATION CORRECTION FOR LOAD FRAME COMPLIANCE

Due to the load frame compliance of the testing system and the presence of the non-rigid pressure plate force sensor, corrections need to be made to the data. These corrections can be thought of as a calibration for the complete measurement system. The calibration is twofold. The first correction is to the displacement of the sensor, which is related to the measured force. The second correction is to the phase shift of the system.

The displacement correction uses the linear calibration data from Figure 4.8 to correct for the movement of the load frame when a force is applied to the sample tested. The calibration of displacement has to work out how much of the displacement at a given driving voltage is due to the sample, and how much is due to everything else, including the movement of the pressure plate force sensor.

According to the specifications of the PZT system, the relationship between driving voltage, $V$ (V) and displacement, $D$ ($\mu$m) is:

$$D = 5V.$$  \hspace{1cm} (4.5)

From the linear test of Sensor 1 shown in Figure 4.8, it is determined that:

$$F_0 = 47.4V_0 - 0.13$$  \hspace{1cm} (4.6)
This relationship can be used to confirm Equation 4.5. Using a manual micrometer stage the FBG sensor was pressed onto the pressure plate force sensor in steps of 2.5 µm, and the resulting force was recorded. The data was used to create a graph depicting the relationship between force (mN) and displacement (µm). Using Equation 4.6 this graph can be converted to a graph depicting the relationship between displacement and voltage ($V_0$) as shown in Figure 4.10. Fitting a straight line to this graph, the relationship between displacement (µm) and voltage (V) gives:

$$D = mF_0 + c,$$

which is calculated to be:

$$D = 5.1V_0 + 0.2,$$

which is in reasonable agreement with the specification shown in Equation 4.5.

Figure 4.10: Graph of the relationship between voltage and displacement for the FBG sensor system.

To correct the displacement of the sensor when a sample is present, the measured voltage $V_m$ is used to calculate the corresponding force ($F$) (i.e. the peak-peak amplitude of the force measured by the FBG sensor, shown in Equation 4.1). This force, $F$ is then substituted into Equation 4.6, to determine the system offset voltage, $V_0$. The corrected voltage,
$V_c$, which is the voltage required to determine $D_s$ for the sample, is then calculated using the following equation:

$$V_c = V_m - V_0.$$  \hfill (4.9)

Therefore Equation 4.5 can be simplified to:

$$D_s = 5V_c.$$  \hfill (4.10)

This result is then used in Equation 4.1 to determine the storage modulus. This calibration is based on the reasonable assumption that the displacement due to the load frame compliance and the sample add linearly for a given applied force. For the same reason we are able to ignore the displacement due to the preload, as it is straightforward to separate the sinusoidal probing force component from the constant preload (see Figure 4.11).

![Figure 4.11: Schematic representation of the static force $P$, which represents the preload applied to all samples at the start of testing, and the dynamic applied force, $F$.](image)

In order to correct for the phase shift due to the measurement system, or in other words, the dynamic response of the system itself, data was recorded without a sample present i.e. the FBG sensor was in direct contact with the pressure plate force sensor. From this data the phase shift ($\phi_0$) was calculated using Equation 4.2. This phase shift ($\phi_0$) was
then used to determine the true phase shift ($\phi_s$) of the data due to the sample being tested according to the equation:

$$\phi_s = \phi_m - \phi_0.$$  \hspace{1cm} (4.11)

The true phase shift ($\phi_s$) is then used in Equation 4.3. Once again this correction is based on an assumption that the phase shifts due to the load frame compliance and the sample will add linearly. This assumption appears justified in the absence of system resonances, or where the sample generates small perturbations in the phase response.

4.6 REPEATABILITY OF THE FBG SENSOR SYSTEM

In order to understand the viscoelastic properties of the materials tested, the material should be examined over a range of frequencies. There are two ways to achieve this: either continually sweep the frequency over the desired range, or measure at discrete frequencies across a frequency range. The literature suggests that it is common practice to step through a range of discrete frequencies, as is typical in DMA [17, 32]. In order to evaluate the two options, the complex modulus of 10:1 ratio PDMS was calculated using both a swept frequency and at discrete frequencies across a frequency range. These results are shown in Figure 4.12. The swept frequencies were averaged into discrete frequency bins in order to decrease the variability.

Analysis of Figure 4.12 shows that there is an increase in the error in the data when using a swept frequencies approach (average standard deviation = 0.6 MPa) compared to discrete frequencies (average standard deviation = 0.03 MPa). This is particularly noticeable at the lower frequencies. It was noted that there was less data at the low frequencies due to the increased period of each cycle in the linear sweep method used. This would contribute to the increased error. Using a non-linear sweep could help to resolve this issue but was not available on the signal generator used here. As an alternative, the sweep cycle was repeated a number of times, so that there were a significant number of peaks at each frequency in Figure 4.12. The signal was also less stable, possibly due to calibration issues (requirement for regular calibration of PZT system) and there remained an increase in the recorded error for all frequencies, not just the low frequencies. The increased error seen in the swept data could be the reason that discrete measurement frequencies are generally favoured. It is possible to reduce this error by increasing the size of the frequency bins that the data is put into; however this will not remedy the in-
4.6 REPEATABILITY OF THE FBG SENSOR SYSTEM

Figure 4.12: Comparison of the complex modulus of 10:1 ratio PDMS measured both at discrete frequency steps and with swept frequency across the frequency range. The error bars represent the standard deviation calculated from both repeat tests and averaging of data within each data set.
stability of the signal due to the reduced number of data points at the lower frequencies. As a result discrete frequencies across a frequency range were used for all further testing.

In Chapter 3 the issue of variability in the DMA measurement system was discussed. It was important to design a method that is not limited by the same issue. To test the repeatability of the system, seven different 10:1 ratio PDMS samples were tested. These are the same samples that were tested in Chapter 3. It was determined that the standard deviation of these samples was 0.03 MPa. This is much improved from the standard deviation found using the DMA, namely 0.55 MPa. If the same experiment was repeated using a single sample the standard deviation was reduced to 0.001 MPa, suggesting that most of the variability in the data from the benchtop setup can be attributed to sample-to-sample variability. The same can not be concluded for the DMA where the single-sample standard deviation was 0.4 MPa.

4.7 Dynamic Mechanical Properties of Bulk Samples

4.7.1 Samples

Bulk PDMS samples were used to test the benchtop setup, to determine if the FBG sensor could make a distinction between the different complex moduli of the samples. The ratios of silicone elastomer base and silicone elastomer curing agent tested were: 10:1, 20:1, 40:1 and 50:1. These samples were the same samples as used in Section 3.2.

4.7.2 The Effect of Preload on the Dynamic Mechanical Measurements

During testing all samples were preloaded, with a static force applied to the sample before the sinusoidal force (with driver offset) was applied, as shown in Figure 4.11. Please note that the offset sinusoidal drive signal was used because the PZT is only designed for positive input voltage. The preload is important as it makes sure that the FBG sensor does not lose contact with the sample during testing.

Du et al. [17] found that the amount of preload applied to the sample had an effect on the complex dynamic modulus measured. This finding was tested by applying four different preloads to a 10:1 ratio PDMS sample and evaluating the resulting complex moduli. In this thesis the preloads tested were: 20 mN, 30 mN, 40 mN, and 50 mN. A
positive 5 V peak-peak sinusoidal signal was then applied on top of this. These results are shown in Figure 4.13.

![Figure 4.13](image_url)

Figure 4.13: Data showing the effect of preload when measuring the complex modulus of a 10:1 PDMS sample. The error bars represent the standard deviation calculated from both repeat tests and averaging of data within each data set.

The results show that preload does have an effect on the complex dynamic modulus measured for the sample. As the preload increases, so too does the recorded modulus. When larger forces are applied to the PDMS sample, such as increasing the preload, there is a larger deformation of the sample. The effect of preload on the modulus could then be attributed to the large deformations contradicting the small deformation assumption in linear viscoelasticity theory. Furthermore, the theory assumes that the sample is an infinite half plane. In this case, as the preload increases the indentation of the sample caused by the sensor approaches the size of the sample and therefore the sample no longer approximates a half plain. This is particular relevant as the edges of the sample are unconstrained. The rate at which the modulus increases with increasing preload is seen to decrease. The average percentage increase is 15.86% between 20 mN and 30 mN preload, 8.2% between 30 mN and 40 mN preloads and 5.7% increase between the 40 mN and 50 mN preload. There are also some differences in the percentage differences
4.7.3 Comparison of FBG Sensor and Pressure Plate Force Sensor

When determining the dynamic complex modulus using the benchtop setup, data was recorded using both the pressure plate force sensor and FBG sensor at 50 mN preload. These results can be compared to make sure that the FBG sensor is measuring force correctly. Data from the pressure plate force sensor and FBG sensor for 10:1 ratio PDMS are plotted on the same axes in Figure 4.14. The result shows good correlation between the two data sets with a calculated $r^2$ value of 0.995. This $r^2$ value was calculated by plotting the force recorded using the pressure plate force sensor against the force recorded using the FBG sensor and fitting a straight line of best fit to the result using the Matlab curve fitting tool (cftool).

4.7.4 Complex Modulus Results for Bulk Samples

The data obtained for the bulk PDMS samples at different ratios is shown in Figure 4.15. The results depict the FBG sensor’s ability to measure different complex moduli. These results are similar in magnitude to the results published in the literature using a similar method including the results shown in Figure 3.1 [22, 15, 17]. The results in the literature give the modulus of 10:1 ratio PDMS to be approximately 1 MPa, whereas results from the system developed in this work show an average modulus for the 10:1 ratio PDMS to be 6.5 MPa, but is estimated to decrease to 4.4 MPa at 0 Hz (static). Although of the same magnitude, this value is larger than the reported values. This result is somewhat
Figure 4.14: Comparison of data obtained using the pressure plate force sensor and FBG sensor for 10:1 ratio PDMS. The error bars represent the standard deviation calculated from both repeat tests and averaging of data within each data set.
expected, as the preload used here is much higher than that reported in Du et al. (50 µm compared to about 1.25 mm for 50 mN used in this work)[17]. It has been shown both in Figure 4.13 and by Du et al. [17] that increasing the preload increases the recorded results. Therefore this could be the reason for the increase in the results for 10:1 ratio PDMS shown in Figures 4.13 to 4.15.

It has also been reported that the measurement technique and test method can affect the recorded results [15, 18]. Although the benchtop test method takes theory from other dynamic mechanical analysis methods, it is a new approach. For example in the benchtop system the test sample is compressed using an FBG sensor, instead of compressing the whole sample and there is no furnace or sample clamping used. It is therefore plausible that this in combination with the increased preload and sample size [163, 18] could also contribute to the difference seen in the results for complex modulus. Although the elastic modulus of 10:1 ratio PDMS is often reported around 1 MPa [15, 22], variations in reported data were found to include elastic moduli as low as 60 kPa [18] and as high as 2.6 MPa [163]. Variations in these results may be due to differences in the data acquisition method. A greater understanding of the methods used to determine these values, including calibration methods are needed to be able to better compare values found using different methods to determine the complex Young’s modulus (E’)[18].

The results shown in Figure 4.15 as well as all the results shown in this chapter are for the restricted frequency range of 0 - 20 Hz. This restriction was due to the limitations of the frequency response of the feedback control system. It is possible that testing over an increased frequency range could provide more information. It is also possible that the recorded average complex modulus could be different if testing was completed over a wider or different frequency range; this is due to the frequency dependence of the material properties.

Figure 4.15 also shows the real and imaginary moduli of the different ratios of PDMS. It is possible to imagine two different materials that may have the same absolute value of the complex modulus. In this situation it may be possible to use information contained in the real and/or imaginary moduli to differentiate between the two materials. In the case of the PDMS shown in Figure 4.15, there is little difference between the 40:1 and 50:1 data in both the complex and real modulus. However in the imaginary data there is an increase in the separation between the two data sets, especially at the higher frequencies. In fact over the frequency range tested there is only a 18.2% difference in the 40:1 and 50:1 ratio PDMS values when comparing the absolute value of the complex modulus, this increases to 40% when comparing the imaginary modulus alone. It is therefore possible
4.7.5 Layered PDMS Sample

A layered PDMS sample was also tested to explore the FBG sensor’s ability to resolve spatial variations in stiffness. This sample was designed to determine if the FBG sensor is capable of identifying changes in tissue properties at the mesoscale. This sample was designed to have layers of 10:1 and 50:1 ratio PDMS. The sample was tested along a line perpendicular to these layers as shown on the left hand side of Figure 4.16.

4.7.6 Making of Layered PDMS Sample

The layered PDMS sample was made using 10:1 and 50:1 ratio PDMS. The sample was cast in a petri dish with approximately 1 mm thick layers. There were five alternating layers in total, with 10:1 ratio PDMS forming the top and bottom layers. The sample was made one layer at a time. The first layer was poured and degassed and then placed in the oven for four hours. The sample was then removed from the oven and the second layer was poured on top. The layer thickness was controlled by adding a constant mass of liquid PDMS to each layer. The second layer was also degassed and placed into the oven overnight. This process was repeated until all five layers were in the dish. Please note that each successive 10:1 layer was placed in the oven for 4 hours and the 50:1 were baked overnight. The oven temperature was kept constant at 80°C. The sample was then left for a week to settle at room temperature. This is consistent with common PDMS processing conditions. Once settled, a small cube was cut out so that testing could be completed across the sections with different stiffness as shown in Figure 4.16. It was important to get the cut as smooth and as straight as possible to reduce the impact of surface irregularities on the measurement. The cutting was done with a custom-made mitre box and a blade. The sample obtained is shown on the right hand side of Figure 4.16. Some unavoidable cutting artefacts (mainly horizontal striations) are visible in the image.
Figure 4.15: Comparison of results for different ratios of PDMS showing: top) the absolute value of the complex modulus; middle) the real modulus and; bottom) the imaginary modulus. The error bars represent the standard deviation calculated from both repeat tests and averaging of data within each data set.
4.7.7 Complex Modulus Results for Layered PDMS Sample

Having demonstrated that the FBG sensor can determine different stiffness properties the layered PDMS sample was used to test whether it can spatially resolve changes in stiffness. The results of this experiment are shown in Figure 4.17. In order to confirm the repeatability and stability of the measurement system, the sample was tested starting at one side of the sample and then the other. The real and imaginary moduli for one of the directions are shown in Figures 4.18 and 4.19. The sample was also rotated 180° with respect to the measurement direction and the process repeated. These results are shown in Figure 4.20. All data was recorded at 10 Hz, in the middle of the frequency range used for the bulk samples.

It was predicted that the results for this sample would be a step function with peaks at the modulus recorded for the bulk 10:1 ratio PDMS and troughs at the modulus recorded for the bulk 50:1 ratio PDMS. The dotted lines in Figures 4.17 to 4.20 represent the values for the bulk PDMS samples at 10 Hz, for reference. The results of the layered PDMS sample (Figure 4.17) show more of a sinusoidal shape, with the peaks and troughs increasing in value as the horizontal measurement position moves across the sample. The peaks and troughs are also at different values than expected. In order to ensure that the increasing trend in the peaks and troughs was not due to hysteresis in the system, the measurements were repeated by reversing the measurement direction (Figure 4.17) and also by rotating the sample by 180° (Figure 4.20). The results were reasonably similar in

Figure 4.16: Schematic (left) and image (right) of the layered PDMS sample, containing alternating layers of 10:1 and 50:1 ratio PDMS.
Figure 4.17: Dynamic modulus of layered PDMS sample recorded by translating the FBG sensor in both forward and reverse directions. The black dotted lines represent the values corresponding to the complex modulus of the bulk PDMS samples. The error bars represent the standard deviation of the recorded data set.
Figure 4.18: Real modulus of layered PDMS sample, shown with the values corresponding to the real modulus of the bulk PDMS samples. The black dotted lines represent the values corresponding to the real modulus of the bulk PDMS samples. The error bars represent the standard deviation of the recorded data set.

Figure 4.19: Results of the imaginary modulus of the layered PDMS sample. The black dotted lines represent the values corresponding to the imaginary modulus of the bulk PDMS samples. The error bars represent the standard deviation of the recorded data set.
Figure 4.20: Dynamic modulus of layered PDMS sample recorded in one direction, then the sample is flipped (180°) and retested. For comparative purposes the 180° sample data has also been flipped 180° after processing so that both data sets appear to be in the same direction. The black dotted lines represent the values corresponding to the complex modulus of the bulk PDMS samples as shown in Figure 4.17. The error bars represent the standard deviation of the recorded data set.
the overall trend and shape, ruling out any potential hysteresis, misalignment or other sorts of experimental artefact i.e. the variation is due to the sample, not the measurement technique. To further test this theory, a shear force was manually applied to the sample as shown in Figure 4.21. In the image it can be seen that there is a decrease in the amount of sample deflection across the sample. This suggests that the stiffness of the sample increases across the sample when moving from the final cast layers through to the first layer. This is consistent with the recorded results.

![Image of shear force being manually applied to the layered PDMS sample. This shows increased stiffness of the sample as you move from left to right.](image)

In Figure 4.17 it can be seen that the data moves away from the dotted lines representing the 10:1 and 50:1 ratio PDMS bulk samples. This suggests that the layers are interacting with each other. The presence of the different stiffness layers may have an effect on the results, similar to a composite material. As discussed above, Figure 4.17 also shows a drift in the recorded modulus as the horizontal position moved towards the deeper cast layers of the sample. The upward drift in the modulus as the sensor moved across the sample could be due to the manufacturing process. As mentioned previously, the sample was constructed by layering the different ratios of PDMS. The sample was placed in the oven once each new layer was added. This process resulted in the bottom layers being in the oven longer, possibly affecting the overall stiffness of the PDMS. It is also possible that some diffusion of curing agent occurred between the layers, resulting in the troughs being above the bulk value. The drift in the modulus and the deviation from the bulk values suggest that there has been some diffusion between the layers.

The real modulus for the layered PDMS sample shown in Figure 4.18 is similar to the results for the complex modulus (Figure 4.17). However the imaginary modulus shown in Figure 4.19, shows more values outside the expected range. This suggests that the
layered PDMS sample, which can be considered a composite material, behaves differently to the individual components. There is a need for finite element modelling to better understand the characteristics of combined materials and heterogeneous tissues in complex biological structures.

Finite element modelling of composite materials could be used in the future to gain a better understanding of what is happening here. Although the overall trend of the results in Figure 4.20 are consistent, there appears to be an increasing offset between the measurement as the probe moved across the sample. This could be due to the buckling and bending of the FBG sensor that was observed when trying to record data at the boundaries between layers, potentially causing increased error in the measurement position. While the precision of the horizontal stage movement is very high, the actual position error of the fibre is higher.

Some outliers (spikes) were seen in the data. These outliers appear to be located near the boundaries between the layers. A 3D optical profile image was also taken of the surface of the sample, shown in Figure 4.22. This image shows changes in the height of the surface. Therefore the outliers could be due to the FBG sensor getting wedged or deflected into the deeper valleys between the layers of the PDMS causing the FBG sensor to buckle. It is believed that the changes in height are due to the cutting process. When the blade moved through the sample the softer parts of the sample were compressed. Fortunately the change in thickness across the sample is much less than the indentation due to the preload applied to the sample before testing, therefore having minimal effect on the overall result. Due to the difficulty in cutting the sample a different manufacturing method for this kind of test piece would be preferable in the future.

Setting aside the complexities of the sample structure, the results confirm that the FBG sensor is able to determine changes in stiffness properties on a scale of approximately 0.5 mm. This scale is consistent with the goal of probing the mesoscale, which is a desirable measurement scale for determining potential detrimental changes to tissue stiffness (see Chapter 1).

4.8 Conclusion

The benchtop setup has proved to be a useful tool in testing the theory that an FBG sensor can be used to determine the dynamic mechanical properties of materials. The FBG sensor was able to measure the difference in the complex modulus of PDMS samples with four
Figure 4.22: 3D profiler image of the surface of the layered PDMS sample, showing the different heights in the sample. The length of the scanned region is 5 mm, while the colour-coded vertical scale is measured in micrometers.
different stiffnesses, as well as measuring the spatially dependent changes in stiffness in a sample of varied stiffness. These results suggest that the FBG sensor is a promising tool for determining differences in the stiffness properties of viscoelastic materials in the mesoscale. While the results obtained from the FBG sensor show good repeatability and satisfactory precision, the results were observed to differ quantitatively from previously published data for PDMS. This difference appears to arise primarily from the different level of preload applied to the samples. For example, the DMA applied a preload pressure of 5.7 kPa (see Section 3.3.1), while the FBG sensor was used with a preload pressure of 4.1 MPa. This relatively high preload was primarily required to improve the signal-to-noise ratio of the sensor read-out. Therefore in future it will be of interest to improve the sensitivity of the sensor in order to allow smaller preloads and smaller force amplitudes to be used. This might also serve to further reduce the spacial resolution of the measurement, which is currently estimated to be about 0.5 mm. Future work should seek to compare the measured results against finite element modelling, in order to understand how the composite properties of the different components influence the measurement. Improved methods are required to fabricate samples with well controlled and predictable properties.
DEVICE DEVELOPMENT: ENDOSCOPE COMPATIBLE DEVICE TO DETERMINE DYNAMIC PROPERTIES

This thesis has so far focused on developing a method for measuring the changes in dynamic mechanical properties using an FBG sensor. To take this from a laboratory-based concept to a more clinically-relevant technology, a portable driver device must be designed. For biomedical applications this driver device must be developed to press the FBG sensor onto the tissue being tested over the frequency range required, in order to allow tissue measurements in vivo. The benchtop system used a piezoelectric transducer to achieve this in the laboratory setting. A piezoelectric transducer is not considered to be appropriate for use in vivo as it requires relatively high voltages and requires a complex feedback system to ensure linearity and are expensive to produce. It is possible that given the development of an appropriate driver, this technology could be used in a number of clinical applications including characterising oral tumours, measuring brain tissue, stiffness measurements during endoscopic surgery and gaining a better understanding of tissue properties for 3D printing and other applications. It is also possible that the design of the driver would differ slightly depending on the application. Endoscopic surgery was chosen as a starting point for design, as it is a minimally invasive technique and could be commonly used. This chapter focuses on the design and development of a driver prototype that would allow the FBG sensor to be used as a portable tissue stiffness measurement device that is endoscope compatible.

5.1 REQUIRED ACTUATOR SPECIFICATIONS

Many conditions including cancerous tumours, that involve changes in tissue stiffness properties, are diagnosed using endoscopic exploration. Endoscopic exploration is commonly used to investigate changes in the tissue of the bowel, stomach, nose, mouth, lungs
and throat. Endoscopic exploration allows medical professionals to visually explore the tissue in real time. An FBG sensor that can determine the dynamic stiffness properties of the tissue during endoscopic exploration will allow the medical professional to have more information at hand, aiding in better diagnoses of potentially detrimental changes to tissue health. In order for the FBG sensor to be endoscope compatible it must be small enough to fit into the instrument channel of an endoscope, which is commonly 3 mm, but can range from 1 to 5 mm, depending on the type and size of the endoscope [164]. Any part of the FBG sensor that enters the body must be made from biocompatible materials, it must also be a disposable component or be able to be sterilised. It is also important to make sure that the driver complies with all the electrical safety regulations for medical devices. As this thesis focuses on the development of a proof-of-concept prototype, much of the finer details have not been fully considered at this time. However it is important to understand that these things are important in the final design of the FBG sensor.

Based on the current performance of the FBG sensor (see Chapter 4), the actuator of the FBG sensor system should press the FBG onto the tissue with a small constant amplitude of about 25 μm. This provides the displacement $D_s$ in Equation 4.1. The driver should have the ability to apply a preload to the tissue of about 50 mN before the driving force is applied, to make sure that the FBG sensor is in constant contact with the tissue being tested throughout the testing process. The driver should be able to drive the FBG sensor onto the tissue at a variety of frequencies. The benchtop testing rig for the FBG sensor was capable of testing the method in a frequency range of 0-20 Hz. The driver should be able to cover this range at a minimum but preferably would be able to extend the range above 20 Hz to about 100 Hz, where feasible.

At this stage the issue of load frame compliance is ignored, but would need to be addressed. The problem of how to hold the sensor firmly in position while performing the measurement is likely to be a key challenge in interpreting the results of such a device.

5.2 DESIGN OVERVIEW

When designing the driver for the FBG sensor a number of options including a PZT, electromagnetic actuators and a hydraulic driver were considered. All methods have their limitations. With regard to the PZT method there was difficulty obtaining a PZT that is compatible with the size of the endoscope instrument channel. It is however possible to
use a large PZT or simple electromagnetic actuator such as a speaker driving a longer length of fibre. The next issue with PZTs is the feedback system. Feedback systems are required to control the reliability of operation. However they also increase overall complexity and cost. Although the feedback system used in Section 4.4 was not ideal, experience was gained into some of the possible difficulties, including instability and restriction of the frequency range over which measurements can be taken. During the benchtop testing completed in Chapter 4, it was found that after each recalibration of the feedback system, the frequency range over which measurements could be taken without large amounts of noise changed slightly.

As alternatives to PZTs, the other methods investigated also have their limitations. The electromagnetic force method is complicated. Hydraulic drivers may be susceptible to significant frictional damping of the applied force. This chapter explores using a number of actuator types to assess their suitability to perform as a driver for an endoscope compatible FBG sensor to measure the dynamic mechanical properties of tissue.

5.3 Design Based on Speaker Coil

5.3.1 First Generation Speaker Coil Actuator Design

The initial design aimed to test the possibility of propagating the oscillatory force produced by a speaker down a length of optical fibre. This was to test the likelihood of designing a system where the oscillatory force is produced by a device above the endoscope and propagated via optical fibre to the tissue. The testing set up was as follows: a capillary tube with an optical fibre threaded through the centre, with one end of the optical fibre glued onto a speaker. The inner diameter of the capillary tube was approximately 300 µm. This setup is depicted in Figure 5.1. The optical fibre used was unstripped 125 µm SMF-28 (same fibre used in the mechanical testing). The coil and fibre were driven by a square wave. A sinusoidal wave form will be required for the final version of the FBG sensor system, but a square waveform is sufficient, as a proof of concept. The frequency and amplitude of the square wave could be adjusted. In order to test the system, a small piece of optical fibre was left protruding out of the other end of the capillary tube. This end was placed against the pressure plate force sensor (Mark-10 51 – same as other tests) with preload. Data from both the pressure plate force sensor and the driver were recorded.
The capillary tube was used to try to prevent the optical fibre from bending. Equation 2.7 states that as the length of optical fibre increases, smaller forces are required to buckle the fibre. It was hoped that the capillary tubing would reduce the effective length of the fibre; hence the buckling force would be calculated using the length of the fibre protruding from the capillary tube, instead of the full length of the optical fibre.

Results of the first generation speaker coil actuator design were mixed. The amount of preload was important to reduce ringing around the sharp edges of the square wave. Initially no response was achieved as seen in Figure 5.2. The pressure plate force sensor data (red) appears to be just noise. It is believed that this is the result of the fibre’s inability to propagate the driving force with enough amplitude for the sensor to record. The loss of amplitude could be caused by friction between the capillary tube and the optical fibre at points of buckling. Significantly increasing the preload (90 mN) and keeping the driving frequency below 9 Hz, a square response was achieved, shown in Figure 5.3. The response data (red) follows the driver signal with a relatively small amplitude. The loss in amplitude could be attributed to the same factors seen in Figure 5.2, but to a lesser extent. Keeping the preload constant, once the frequency is increased to about 130 Hz (Figure 5.4) there is a sinusoidal response that is out of phase with the driving signal. This could again be due to frictional losses or it could be associated with a vibrational resonance of the system.

5.3.2 Second Generation Speaker Coil Actuator Design

Updates from the first generation include changing the drive signal to a sinusoidal wave and replacing the optical fibre with a larger diameter fibre that is stripped. The reason for
Figure 5.2: Results from the first generation speaker actuator at low driving amplitude (frequency is about 20 Hz). The blue line represents the driving signal. The red line represents the response recorded by the pressure plate force sensor.

Figure 5.3: Voltage response from the first generation speaker actuator at higher drive amplitude. The blue trend represents that of the driving signal. The red trend represents the response recorded by the pressure plate force sensor. This was achieved with high preload and low frequency (90 mN and 9 Hz).

Figure 5.4: Sinusoidal voltage response from the first generation speaker actuator. The blue trend represents that of the driving signal at 130 Hz. The red trend represents the response recorded by the pressure plate force sensor.
changing the fibre was that there was a concern that the fibre was buckling with multiple bends inside the capillary tube. Having a tighter fit would help to reduce this. The jacket was removed to try to reduce friction. Initial testing suggested that this version was providing a more faithful transmission of the displacement across the frequency range of interest. However, removing the jacket caused the fibre to become very brittle, and as a result no useful data was able to be recorded with this generation, due to breakage. Given the challenges encountered around friction, it was concluded that this approach is unlikely to be effective if the capillary tube is bent, as would occur in an endoscope system. Therefore work moved toward the next generation of the actuator focusing on a hydraulic actuator.

5.4 Design based on hydraulic actuator

The third generation of the actuator was based on a hydraulic system. This design was based on movement seen in preliminary trials when threading an optical fibre through a medical syringe and using a syringe driver to provide a force. In the subsequent test system, the optical fibre was encased in a tube with hydraulic fluid and a membrane at each end, as shown in Figure 5.5. The hydraulic system was made up of clear silicone tubing with swagelok and customised fittings to hold the piston and actuator membranes. A force was applied to the top membrane causing it to be displaced. This displacement propagates through the fluid, hence moving the second membrane and the attached optical fibre. Testing was completed using different membranes and mounting configurations to test the suitability of a hydraulic system to propagate the oscillatory force, with reduced loss of propagating signal compared to the earlier generation systems.

The aim of the hydraulic actuator was to thread the FBG sensor through the hydraulic system and use an incompressible fluid to propagate the force. A hydraulic system would in principle allow for the FBG sensor probe to be narrow enough to fit into the instrument channel of the endoscope. The type of hydraulic actuator developed to test the concept is shown in Figure 5.6. The fibre was threaded through the right-hand-side of the hydraulic actuator. There was a membrane on the left hand side where the force was applied. There was also a membrane at the base responsible for the movement of the FBG onto the tissue being tested. The stiffness of the membrane is important; it is related to the amount of movement achieved with a particular force. A number of membranes were tested; the
Figure 5.5: Schematic of the design of hydraulic actuator.
Figure 5.6: Left: Image of hydraulic actuator system. Right: Image of hydraulic actuator mounted for testing. The splint is added to help keep the actuator system straight in an attempt to improve transmission of force by preventing adhesion between the fibre and the tubing.
membrane that gave the most amount of movement was used. This membrane was made from the shaping balloons that are commonly used for balloon modelling.

This system was tested by plotting the displacement applied to the membrane against the force recorded on the pressure plate force sensor. This represents the relationship between the applied force and the movement of the FBG sensor. These results are shown in Figure 5.7. Ideally there would be a linear relationship between the force and displacement. It would also be ideal to see forces up to at least 30 mN. Unfortunately as can be seen in Figure 5.7 this was not the case. Not only was there a poor linear relationship, there was also poor repeatability and small forces generated. It was thought that much of this was the result of the fibre sticking to the side of the tubing. This was because the wet fibre was sticking to the inner wall of the tubing, generating considerable drag. In addition the system was not 100% water tight, creating bubbles that damped the hydraulic pressure. Although great care was taken to remove these issues including changing the mounting configuration and refilling the tubing, this issue could not be resolved. Therefore it was concluded that it was no longer viable to continue working on this design.

![Figure 5.7: Data from the testing of the hydraulic actuator showing lack of repeatability due to friction between fibre and tubing.](image-url)
5.5 Design Based on a Voice Coil Actuator

The design of the fourth generation actuator was focused around a hollow core linear actuator (H2W technologies NCM02-05-005-JJB). This compact actuator (13 mm diameter) could provide movement at the required frequencies using electromagnet drive coils and jewelled bushings. This actuator was used as a proof of concept as it was readily available commercially. It is possible to imagine the use of a smaller version of this device which is compatible with the instrument channel of an endoscope (about 3 mm). The hollow core allows for the FBG sensor to be threaded through the core of the actuator and secured. Some modifications to the actuator were made to secure the fibre and to help smooth its movement. Figure 5.8 shows the final configuration of the actuator. The actuator was attached closer to the tip of the optical fibre, reducing the loss of driving signal that was found in previous designs. Collars were custom made to fit on each side of the actuator shaft, and these secured springs that were added to the actuator to help control its movement. The bottom collar also held a glass ferrule to which the FBG sensor was secured before being passed through the centre of the actuator. The glass ferrule was used to hold the optical fibre securely within the hollow shaft so that it didn’t slip or buckle. The shaft of the voice coil actuator moves against the force of the springs when a current was applied. As the optical fibre was secured within the shaft, the fibre and the shaft move together, as shown in Figure 5.9.

Again, the actuator was initially tested by looking at the linearity of the system. To do this, an increasing current was applied to the actuator and the resultant force was recorded. These results can be seen in Figure 5.10. Figure 5.10 shows a good linear relationship between the current and force during both application of force ($r^2 = 0.988$, 0.15-0.4 A), and recoil ($r^2 = 0.999$, 0.3-0.005 A). Figure 5.10 also shows good repeatability between the three repeated tests presented here. However, some hysteresis (difference between loading and recoil is apparent). This could be corrected by the addition of a feedback system or an electrical offset. The importance of a feedback system was discussed in Section 4.4.2, it was therefore not surprising to see this result in the absence of a feedback system. If the actuator was to be used as the actuator for the FBG sensor a feedback system or some form of electrical offset would need to be developed. This was beyond the scope of this thesis, but will be important work for any further research.

This actuator system was further tested to determine if it was capable of replicating the results seen in the benchtop setup. The actuator mounted as seen in Figure 5.8 was very
Figure 5.8: Image of voice coil actuator used as part of fourth generation actuator design. Right: The actuator when it was mounted for testing.
Figure 5.9: Schematic of voice coil actuator used to drive the optical fibre sensor.
Figure 5.10: Graph of the relationship between applied current and force for the voice coil actuator. Each number represents a repeated test. The green line shows the best linear fit.
similar to that used in the benchtop testing (refer to Chapter 4 for more information). Testing was completed on 10:1 ratio PDMS sample, at 5 Hz. Preload was applied to the sample before the driving force was applied, this was consistent with testing in Chapter 4. The initial results shown in Figure 5.11 were disappointing. There are three main issues with the data shown in Figure 5.11: the wave form was square instead of sinusoidal like the driving signal, the data was not symmetrical and there was ringing in the response signal. It was believed that the square wave form was caused by a limit in the force generated by the actuator or the hysteresis shown in Figure 5.10, where the current peaks and turns, but the force stays constant. According to the Lorentz force law \[161\] force is proportional to current. To try and eliminate this issue the current of the driving signal was increased. These results can be seen in Figure 5.12. It can be seen that as the driving amplitude was increased so to did the response, however unfortunately as the driving amplitude was increased, the travel distance of the actuator also increased. This made it impossible to keep the FBG sensor in contact with the sample during testing. Increasing the preload can help to keep the FBG sensor in contact with the sample, but this was limited by the maximum force range of the pressure plate force sensor used. Stiffer springs where added to try and reduce the amount of movement. The springs used were unable to dampen the movement enough to correct this issue.

The poor symmetry of the results was thought to be the result of imbalances of the springs. When initially setting up the actuator, it was determined that at times the actuator would stop moving and could be started again with a push and at times it appeared to be spasmodic in its movement. The addition of the spring helped with this, but further improvement was needed.

The addition of springs into the system was important to aid in the consistency of movement and to reduce the overall amplitude of the travel. However the presence of the springs creates its own problems. As a result the springs used need to be carefully considered. Ringing can be seen throughout the data in both Figures 5.11 and 5.12. Figure 5.13 shows a zoomed in view of the ringing seen at the peaks. The frequency of the ringing was calculated using the same method used in Chapter 4 (Equation 4.4) and found to be \(28 \pm 1\) Hz. This frequency can be used to determine the spring constant \(k\) of the springs used in the system \[161\]. This result can then be compared to the calculated value of spring constant for the desired spring. The resonance frequency of a spring is:

\[
 f = \frac{1}{2\pi} \sqrt{\frac{k}{m}}, \quad (5.1)
\]
5.5 Design Based on a Voice Coil Actuator

Figure 5.11: Results of the initial testing of the voice coil actuator. Testing conditions: 10:1 PDMS, 5 Hz. The blue line represents the driving signal. The red line represents the voltage measured by the pressure plate force sensor (analogue output).

Figure 5.12: Results of actuator testing using increased current to drive the voice coil actuator. The blue line represents the driving signal. The red line represents the voltage measured by the pressure plate force sensor (analogue output).
where $k$ is the spring constant and $m$ is the mass of the moving parts of the actuator assembly.

The mass of the springs and the nuts were measured using a 3 point laboratory mass balance (China Electrical Analytical Balance - JA2603B) and found to be 3.2 g, this combined with the estimated mass of the shaft of the actuator (based on density and volume), the mass of the moving parts are estimated to be about 12 g. The mass of the shaft could not be measured precisely as the shaft cannot be removed from the body of the device. The resonance frequency was measured to be 28 Hz. Using these values the spring constant was estimated to be 370 N/m. According to Hook’s Law [161]:

$$ F = 2kx, \quad (5.2) $$

where $F$ is the force and $x$ is the displacement. Please note in this case the 2 represents the presence of two springs.
Using the values for force and displacement used in Chapter 4, to determine complex modulus, the ideal spring constant can be calculated. This means allowing the driving force to be 250 mN and the displacement to be 25 µm. The desired spring constant can then be calculated to be 5,000 N/m. This is over 13 times larger than the calculated value for the springs used in the current setup.

Using careful selection of springs it is possible that the actuator will prove to be a suitable actuator for an endoscope compatible device to measure the dynamic mechanical properties of tissue. The presence of appropriate springs would allow the driving current to be increased while reducing the amplitude of movement to a specified level. This should result in the desired sinusoidal response assuming that it also serves to reduce the hysteresis. Using Equation 5.1 it was predicted that as the spring constant increases so too does the resonant frequency of the spring. As measurements will only be taken at low frequency (0-100 Hz) the ringing should be quickly damped out in this frequency range, not effecting results. With respect to the third issue; poor symmetry due to stickiness in travel, this too could be corrected with the inclusion of the right springs and perhaps some lubrication (the only lubricant suggested by the manufacture is mineral oil). Improved balancing of the top and bottom springs could also help. Similar to the feedback system, optimisation of the actuator driver system was outside the scope of this thesis; however the results to date are positive and should provide a solid platform for any future research.
CONCLUSION

6.1 INTRODUCTION

Measuring dynamic mechanical properties of biomedical materials in the mesoscale could help to provide vital information about the materials tested. This knowledge could be used as a diagnostic tool or to help build better artificial tissues and phantoms. This thesis investigated the possibility of whether or not an FBG sensor system could be used to determine dynamic mechanical properties of viscoelastic solids at the mesoscale. The thesis began by looking at the development of FBG force sensors, including the manufacture and calibration, followed by a characterisation of the mechanical properties of FBG sensors, including buckling and bending plus dynamic properties. A common laboratory tool (DMA) was used to gain a point of comparison for the recorded values. A benchtop sensing system including a data analysis program was developed to test if the FBG sensor could measure the dynamic properties of PDMS samples with different stiffness and variability within the sample. The possibility of converting the benchtop system into a medical device was also explored.

6.2 OUTCOMES OF THE RESEARCH

Optical fibre FBG sensors and sensor system were successfully manufactured and calibrated for use as a novel method of determining the complex modulus of viscoelastic solids at the mesoscale over a frequency range of 0-20 Hz.

Mechanical testing of the FBG sensor showed that as the length of exposed optical fibre increased the forces required to buckle and bend the sensor decreased. Testing was completed on three different diameters of optical fibre (50 µm, 80 µm and 125 µm), it was found that as the diameter of the fibre decreased the forces required to buckle and
bend the optical fibre also decreased. These force changes were able to be represented by buckling theory (Equation 2.7) and the derived deflection theory (from [131]). Using these results it can be determined that for a 10 mm piece of exposed optical fibre (length of an FBG sensor) the maximum buckling force would be 210 mN (longitudinal) and the maximum deflection force would be 7 mN (transverse) for a 125 μm diameter fibre. These forces are further reduced as the diameter of the fibre used to create the FBG sensor is decreased. These results enable a better understanding of the forces that can be applied to the optical fibre sensor before it buckles or bends affecting the output of the FBG sensor system. The output of the FBG sensor after the optical fibre was buckled were successfully obtained and concluded to be predictable if the buckling angle is known. The output of the FBG sensor once it has buckled is the cosine of the applied force.

Dynamic mechanical testing was also completed on the FBG sensor using DMA. These results were limited by variability however; average results were similar to those published in the literature. DMA results for PDMS samples at two different base:crosslinker ratios (10:1 and 50:1) were also achieved. Comparison of the DMA results confirmed that the optical fibre sensor is much stiffer (GPa compared to MPa) than the PDMS sample tested. This is a desirable characteristic for the FBG sensor system.

The dynamic mechanical properties (stiffness) were successfully measured for four different base:crosslinker ratios of PDMS (10:1, 20:1, 40:1 and 50:1) using the benchtop FBG sensor system and a developed analysis technique including a Matlab code, and calibration process. The results achieved were similar to those recorded in the literature. These results are shown in Figure 4.15. The FBG sensor system was able to detect small changes in the complex moduli of the different samples, down to about 0.1 MPa, with an average error of approximately 0.05 MPa over the frequency range of 0-20 Hz.

However, these measurements confirmed the dependence of the complex Young’s modulus ($E^*$) on preload and testing technique. It was found that as the preload was increased, so too was the Young’s modulus ($E^*$) determined by the FBG sensor system (Figure 4.13). This is consistent with other findings reported in the literature. It was also found that the measured results are bias of the measurement technique used [18]. Therefore the variations in calculated moduli measured using the benchtop system compared to the results found in the literature could be attributed to differences in the measurement technique and the increased preload used.

The FBG sensor system measured changes within a PDMS sample (Figure 4.17), by measuring the changes in the layered sample due to the presence of different layers of different ratios of PDMS. This showed that the FBG sensor system resolution is compatible
with measurement on the mesoscale (0.5 mm). This testing represents changes in human tissue properties that may be seen in vivo.

The sensor system was also able to reveal information about the layered PDMS sample tested in Chapter 4, that was not immediately apparent after production. The FBG sensor system was able to measure the stiffness gradient of the layered PDMS sample that possibly resulted from repeated baking or cross-linker diffusion.

The specifications required for a medical device that stems from the sensing technique developed in this work were explored and an initial prototype created. These requirements include compatibility with an endoscope instrument channel, the ability to drive the FBG sensor over a frequency range up to as high as 100 Hz, consistent driving amplitude and reliable propagation of the required forces. Of the methods investigated, the voice coil actuator, tested in Section 5.5 was best able to achieve these requirements, making it the most promising of the four candidates tested for the development of a medical device using the FBG sensor technology.

6.3 LIMITATIONS OF THE RESEARCH

Given the time constraints associated with a PhD, there were several areas of work undertaken that could have received further attention if there was more time. The PZT and the amplifier/feedback system were not designed to be paired together. This limited the frequency range at which dynamic mechanical properties could be tested. This meant that all testing was completed between 0-20 Hz instead of the planned range of 0-100 Hz (larger frequency range may provide more information about the test sample). This issue also resulted in the system needing regular recalibration by the supplier in order to achieve repeatable data. In order to avoid this situation in the future, it is important to ensure that the PZT and the feedback system are better matched. This can be achieved by making sure that the notch filter in the feedback system is able to remove the resonance of the PZT in the middle of its tuneable range. A new feedback system was not purchased for this work due to the associated expenses. Testing over a wider frequency range may have provided additional information about the samples tested.

Similarly, testing of the layered PDMS sample was only completed at a single frequency, 10 Hz. Time constraints limited the amount of testing that could be completed on the layered PDMS sample. Testing at a variety of frequencies may have provided some interesting additional information about the sample.
The current FBG sensor system is also constrained by limited force resolution of the sensor. For testing soft materials such as PDMS, this meant that relatively large preload forces and large amplitude oscillations were required to achieve acceptable signal-to-noise ratios. The large preload appears to contribute to the variations observed between the results presented here and those from previous studies. The large preload is also likely to restrict the spatial resolution of the sensor due to shear stresses generated across the area of the indented region. In future work it would be desirable to increase the force resolution of the sensor, according to the principals presented in [155]. Higher force resolution would allow greater flexibility in any future studies of real tissues, as well as for in vivo studies.

The investigation with the turn-key DMA instrument in Chapter 3 highlighted the importance of regular calibration and careful control of environmental parameters to achieve repeatable results. Some concerns were noted about the condition of the DMA. Mechanical factors including the physical characteristics and alignment of the DMA [160, 19] can affect the precision of results. The DMA used in this work is no longer supported by the manufacturer. It has been heavily used over a period of several years without much maintenance or calibration. As a result, some of the variability seen could potentially be due to the use of a sub-optimal device. Use of a newer DMA or a better maintained DMA may have produced more predictable results.

Much learning was required when making up PDMS samples. There were unforeseen issues with achieving consistency in prepared samples and in cutting samples. This resulted in much consultation and remaking a number of samples before testing. PDMS was initially chosen as a test sample as there were a number of papers that compared its stiffness to that of tissue properties [15, 22]. However, after initial testing of the PDMS samples made, it was found that there was variability in the samples making it difficult to make comparison. Insuring the crosslinkers are well mixed, helps to remove this variability within samples made by the one person, however there is questionable variability between different users. Cutting the PDMS also proved difficult. The PDMS sample fabrication process would benefit from further refinement in future, particularly when fabricating samples with mixed properties.

Work was performed on the conversion of the FBG sensor system from a measurement technique to a useable medical device prototype limiting the adaptability of the technique into the medical field. Nevertheless, the work presented here should create a good platform for further research into device development.
As testing moved along it became increasingly clear that the load frame compliance of the testing system plays an important role in good data acquisition. With the benchtop system this issue was overcome by using a large stiff mounting. This level of load frame control may not always be possible with a portable device. This, along with issues of holding the device, applying preload, and stabilising temperature and other environment conditions will need to be further investigated.

This thesis does not include any finite element modelling of the measurement results in order to support the measurement technique. Modelling would allow the method to be further validated as well as providing theoretical validation of the measurement values for viscoelastic materials, thus helping to create a data base of mechanical properties of tissue [165]. In particular, finite element modelling may have allowed further exploration of existing experimental results and a better understanding of the results achieved with the layered PDMS sample. It is possible that there may be increased complications with using this method to determine the dynamic mechanical properties of complex biological structures, and finite element modelling could help to better understand these issues.

Limited data was found in the literature outlining the dynamic mechanical properties of viscoelastic solids, including nylon and polyethylene in addition to PDMS. As there is a lack of published data, and there is variability in the limited data [163, 22, 15, 17], it is possible that further consideration and understanding of the calibration and testing process is required for all methods [18]. Although the test material used (PDMS) has been shown to have properties similar to human tissue [22], its properties have a complicated dependence on hardener mixing ratio, degree of mixing, curing time, humidity and shelf life. As such consistency in production may be hard to achieve [18]. In the future it may be beneficial to test the system with materials that have better characterised mechanical properties (even if their properties are not comparable to the proposed target material of the sensor system). For example, the selected materials could include a purely elastic material and a highly damping material. This would not only help to better confirm the suitability of the FBG sensor system to measure the dynamic mechanical properties of viscoelastic solids, but also help to develop a gold standard for evaluating techniques to measure these material properties.
6.4 IMPLICATIONS OF THE RESEARCH

Dynamic mechanical properties of viscoelastic materials and anisotropic tissues are not well characterised on the mesoscale. The FBG sensor system used in this thesis provides a new technique for measuring these properties. This has been demonstrated using PDMS as a test model. The technique should therefore be able to be used to measure the dynamic mechanical properties of other viscoelastic materials such as human tissue. The development of this new technique will hopefully allow for the dynamic mechanical properties to be measured more readily, helping to gain a better understanding of a wide range of factors affecting tissue (health status, hydration, age) and synthetic materials (hydration, diffusion, fatigue etc). This knowledge could be used as a diagnostic tool or to help create better synthetic tissues and phantoms.

6.5 RECOMMENDATIONS FOR FUTURE WORK

The discussion of limitations of the research in Section 6.3 included a number of recommendations for future work. However, some more general comments are also relevant here.

Further research is needed to create a database of human tissue properties, improve the portable and endoscope compatible device and to find specific applications for the device.

The optical fibre sensor developed as part of this thesis has the potential to be used as a device to characterise the dynamic mechanical properties of a variety of human tissue, hence creating a database of the material properties. Creating a database of human tissue will enable the technology to be used as a diagnostic tool. It is possible to imagine taking measurements of a particular tissue and comparing it to a data base to determine the health of the tissue. Further development of a portable medical device is needed to make sure that the technique is appropriate for its intended use. This will include but is not limited to making sure that the device is endoscope compatible, making sure it complies with medical standards and making sure it complies with electrical safety standards. Completion of the conversion of the design technique into a usable medical device is an important step in taking the technology from a proof of concept to a useable diagnostic device.
Discussions have been initiated with the staff at the Alfred Hospital to possibly use the technology to measure changes in the stiffness of brain tissue to assess traumatic head injuries. Work has also been discussed with the Austin Hospital about the possibility of measuring the stiffness properties of bowel cancer during endoscope investigation, hence the interest in an endoscope compatible device.

There is currently much interest in the biomedical engineering community for the creation of synthetic tissue and tissue phantoms. Measurement of the dynamic mechanical properties of tissue in the mesoscale will help to develop a better understanding of tissue properties so that more realistic synthetic tissues and phantoms can be created.

6.6 Conclusion

The successful measurement of the dynamic mechanical properties of a viscoelastic material (PDMS) in the mesoscale was achieved using an FBG sensor system and custom data analysis technique. This is a fundamental technology that shows great potential for use in a variety of medical applications to help gain a better understanding of dynamic mechanical properties of tissues. Not only is the FBG sensor system capable of measuring dynamic mechanical properties of PDMS at the mesoscale, but should be able to be extended to testing the properties of other viscoelastic materials such as human tissue. The FBG sensor system displayed good measurement repeatability and resolution at the desired scale. This provides exciting scope for further research into the many possible uses of the developed technology.
Part II

APPENDIX
Buckling tests were performed via lowering a force sensor onto the tip of the optical fibre. The testing required the force sensor to be lowered at a set distance and speed. In order to achieve this, the force sensor was attached to a Newport ILS 100 PP translation stage. A Labview program was written to allow the required control of the stage. This section outlines the Labview program created including instructions for operation.

The Newport ILS 100 PP translation stage contains a stepper motor. This stepper motor has a step size (resolution) of 0.5 µm. When writing programs to control the movement of the stage it is worth considering the rate of stepping and rate of sampling. In the case of the buckling experiments the stage was moved at 0.2 mm/s. At this speed you would need a sampling speed of at least 400 Hz. All buckling tests were performed with a sampling rate much higher than this.

A.1 Required Files

There are a number of labview files required to control the stage. Some of these files (operational files) require user input i.e. setting the speed, while others are accessed solely by Labview.

A.1.1 Operational Files

The operational files are the labview files that allow the user to control the movement of the stage via user input. There are three operational files used to control the stage:

- Motor_on.vi
- StartStop.vi
- Motor_off.vi
The motor_on.vi and motor_off.vi files turn on and off the stage driver respectively. The StartStop.vi file allows the user to set the home position of the stage, the velocity of movement and the distance the stage will move. Theoretically it should be able to set the displacement units as well. The default displacement unit is in mm. Much difficulty was encountered trying to change the displacement. The default setting, mm was appropriate for this application, so it was decided that it was not worth spending any more time on this. In the future, the program could be extended to allow for this capability.

A.1.2 Other Files

The operational files require access to a number of other files in order to complete the user input commands. The operational files will be looking for these additional files to be present in the same folder. It is therefore important to make sure that all required files are saved in the same file. The non operational files required are:
- move_distance.vi
- move_to_home.vi
- QD.vi
- read_displacement.vi
- read_microstep.vi
- read_position.vi
- read_res.vi
- read_unit.vi
- set_displacement.vi
- set_home_position.vi
- set_microstep.vi
- set_res.vi
- set_velocity.vi
- stop_motion.vi

A.1.3 Operational Instructions

The below instructions outline the steps to operate the stage as required for the buckling tests.
1. Make sure controller and stage are connected (25-pin sub-D connector – directly connected to the controller)
2. Turn on controller (Newport EPS 300) – on when button pressed in
3. Open motor_on.vi in labview (see Figure A.1)
4. Use drop down box under VISA Refnum in to select controller
5. Repeat process for box under VISA Refnum out
6. Under AXIS type in the number of the axis you wish to turn the motor on (i.e 2)
7. Press the run arrow in the top left of the tool bar (see Figure A.2)
8. Close program
9. Open StartStop in labview (see Figure A.3)
10. Use drop down box under VISA resource to select controller
11. Press the run arrow in the top left of the tool bar (see Figure A.2)
12. Fill in the required parameter and use the adjoining green button to activate the command (click cursor on green rectangle)
13. To turn off motor - Open motor_off.vi in labview (see Figure A.4)
14. Repeat same process as motor_on.vi (steps 4-8)
15. Turn off controller

A.1.4 Clues for Beginners

- Unless instructed otherwise, the controller executes commands in the order received without waiting for completion of the previous command. Commands can be used to prompt the program to wait for commands to finish or to wait for a specified amount of time.
- Command syntax: axisnumber command positionvalue i.e 2VA1 – set velocity of axis 2 to 1. Figure A.5 gives a block diagram of the syntax.
A full list of commands can be found in the manual. Some of the common commands used in this program are shown in Table A.1.
Figure A.1: Image of Motor_on.vi front page. How the program looks when it is first opened.

Figure A.2: Image of run arrow to run Labview programs.

<table>
<thead>
<tr>
<th>Command</th>
<th>Meaning</th>
</tr>
</thead>
<tbody>
<tr>
<td>VA</td>
<td>Set velocity</td>
</tr>
<tr>
<td>TP</td>
<td>Get position</td>
</tr>
<tr>
<td>DH</td>
<td>Define home</td>
</tr>
<tr>
<td>MV</td>
<td>Move</td>
</tr>
<tr>
<td>TP</td>
<td>read actual position</td>
</tr>
<tr>
<td>TV</td>
<td>read actual velocity</td>
</tr>
</tbody>
</table>

Table A.1: Table of commonly used labview commands used to control the stage.
Figure A.3: Image of StartStop.vi front page. How the program looks when it is first opened.
Figure A.4: Image of Motor_off.vi front page. How the program looks when it is first opened.

Figure A.5: Block diagram of the command syntax required to control the translation stage using labview.
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